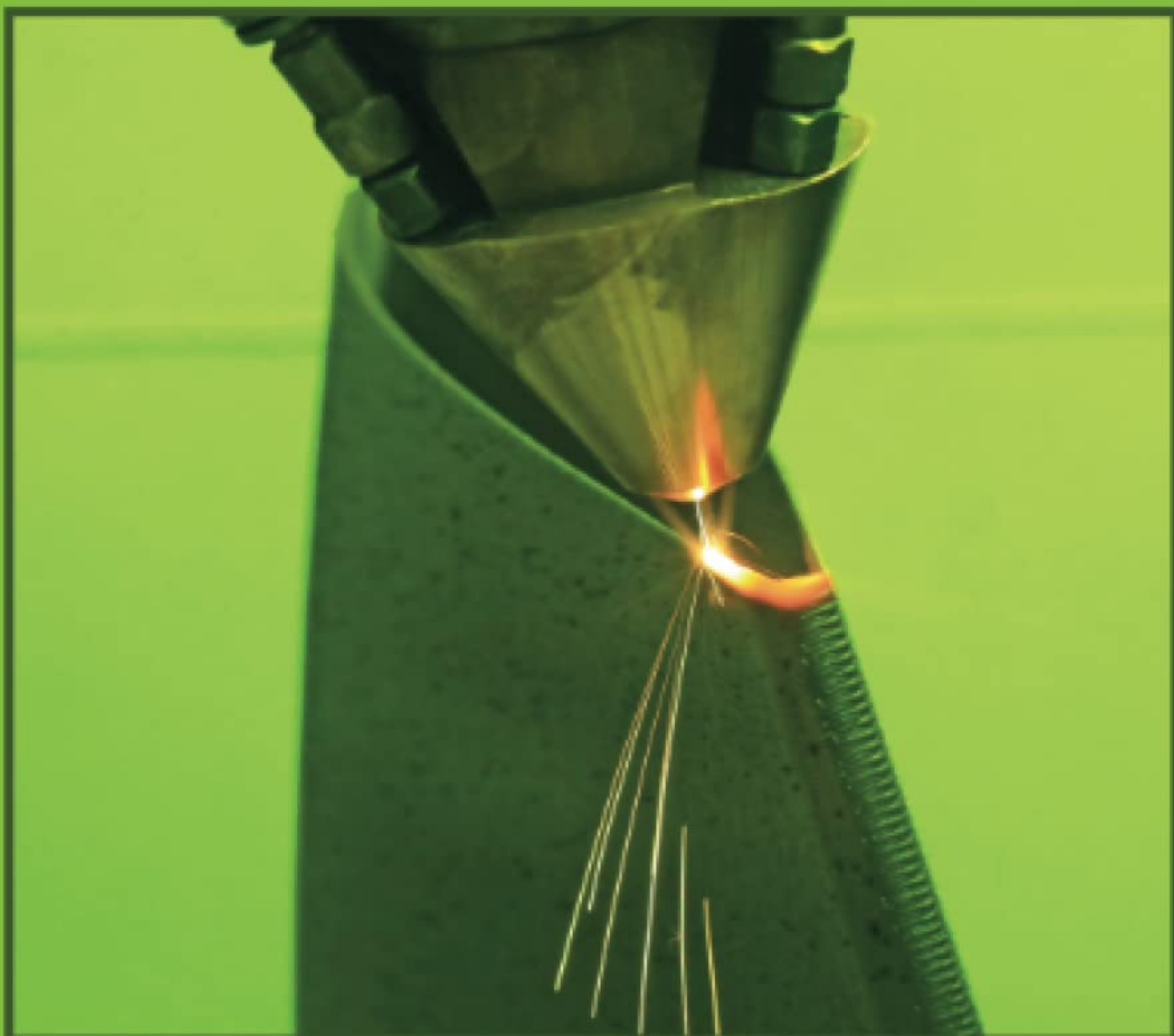


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ADVANCES IN METAL ADDITIVE MANUFACTURING



Edited by
SACHIN SALUNKHE
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Advances in Metal Additive Manufacturing

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Advances in Metal Additive Manufacturing

Edited by

Sachin Salunkhe

Sergio T. Amancio-Filho

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Preface

An essential benefit of additive manufacturing (AM) is the ability to create more complicated parts with better mechanical and thermal performance and lower system mass than is possible with other manufacturing methods. AM design's inherent complexity permits lightweight by consolidating multiple components into one, allowing for increased technical efficacy.

The book is focused on various applications of metal additive manufacturing such as aerospace, defence, automotive, consumer products, industrial products, and medical devices. All chapters of this book present the results of the dedicated research efforts of authors for years. The book highlights the latest research status in not only the domain but also identifies the future scope of work for young graduates, engineers, and senior R&D professionals working in the field of metal manufacturing. There is no doubt that additive manufacturing industries will also be benefited by the research work reported in various chapters of this book.

The book not only highlights the latest research status in the domain but also identifies the future scope of work for young researchers, mechanical and materials engineering students, and professionals willing to learn the fundamentals and recent advances in metal additive manufacturing.

The book provides feature information related to metal additive manufacturing processes such as powder bed fusion (e.g., selective laser melting) techniques, wire- and powder-based direct energy deposition processes using high energy beams, or those hybrid processes that combine both additive and subtractive manufacturing (SM) methods may all fall under the category of AM.

The book's structure provides a guideline of where to start, what to learn, how it all fits together, and how metal additive manufacturing can empower you to think beyond conventional metal processing. In addition, case studies, recent examples, and technology applications are provided to reveal current applications and future potential.

We would like to thank all book chapter authors for their valuable contribution to this book project, and Elsevier and Brian Guerin, Holland-Borosh, Clodagh, and Rafael G. Trombaco for the invaluable help in the organisation of the editing process. Last but not least, the editors would like to thank Rafael Paiotti (TU Graz) for his valuable support with the internal book review activities.

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Powder bed fusion processes: main classes of alloys, current status, and technological trends

1

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1.1 Additive manufacturing of aluminum alloys

The additive manufacturing of aluminum alloys is of high interest for the aviation industry, since the combination of optimized topology and 3D printed parts with lightweight material can lead to a considerable weight reduction, which in turn results in less fuel consumption and CO₂ emission. However, additively manufactured parts for aviation are mostly still not certified to the high standards of the aviation industry, a fact that is expected to change in the coming years. A special challenge in the additive manufacturing of aluminum alloys is the formation of solidification cracks in most alloys due to their bad weldability, which lead to a focus on mostly Al-Si alloys for additive manufacturing in the last years. The following sections will summarize challenges generally for aluminum alloys and common progress in this field especially concerning the laser powder bed fusion (LPBF) technique that is the most prominent one used in this field. A few example alloys and results achieved in these fields will be discussed hereinafter.

1.1.1 General challenges

A general challenge in powder-based methods is the high light reflectivity of aluminum powders in comparison to other common material powders. This leads to a

high necessary energy input for these alloys, which in turn results in longer printing times and higher costs [1–3]. Furthermore, the evolution of different defects as pores and oxides in addition to the phenomenon of balling is also challenging when it comes to processing aluminum alloys. Alloys like AA5024, AA6061, and AA7075, which among other things are very interesting for the aviation industry, are quite prone to heat-cracking due to a tendency to form long columnar grains in the solidification process upon deposition of rapidly heated and cooled layers [4–11]. Fig. 1.1 shows the solidification behavior of these alloys while Fig. 1.2 exemplarily shows the issue of crack formation in an additively manufactured AA5024 alloy. Fig. 1.3 exemplarily shows the two different types of pores of additively manufactured AlSi10Mg. Regarding the morphology of the pores that are formed during the process, one can state that there are mainly two types to be differentiated: irregular pores (Fig. 1.3A) and spherical pores (Fig. 1.3B). While irregular pores are formed due to lack of fusion (e.g., caused by a too big hatching distance, layer height or scanning speed), spherical porosity is caused by gas, which is entrapped in the melt pool during the deposition of one layer. The origin of these gases can be very different, starting with their introduction during the production of the powder or ending with the evaporation of components of the used alloy [12,13].

An additional challenge in the production of aluminum alloys in laser-based techniques operated in a protective gas atmosphere is the oxygen intake during the process, which poses a challenge considering the recyclability of these alloys. Studies have shown that subsequent reuse of aluminum powders can decrease the properties of the material [14–16]. Additional difficulties arise when using laser-based manufactured parts due to the anisotropy of the printed material. Depending on the building orientation, among other things, due to columnar grain growth, mechanical properties of samples tested in parallel and perpendicularly to the build direction can vary greatly [17–19]. Studies at TU Graz have shown a difference in elongation in

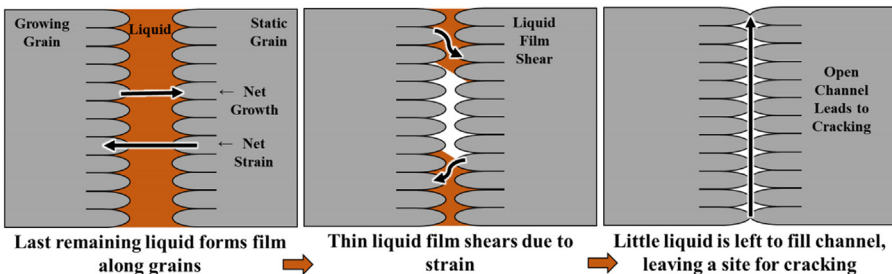


Figure 1.1 The phenomenon of crack formation between columnar grains in additively manufactured AA5024, AA6061 and AA7075 alloys.

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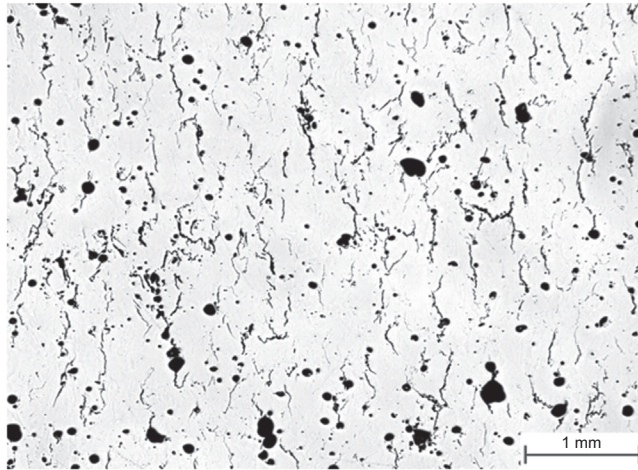


Figure 1.2 The phenomenon of cracking in AA5024 during the manufacturing process (polished surface). Cracks usually appear parallel to the building direction and the direction of solidification of the columnar grains.

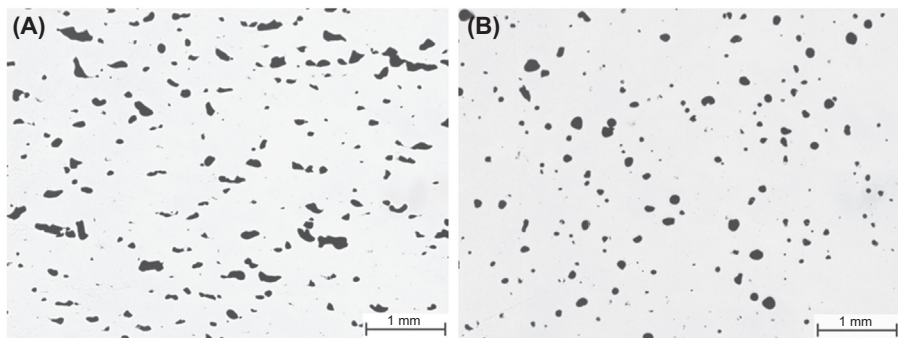


Figure 1.3 Different types of pores in additively manufactured AlSi10Mg. The samples (A) and (B) were manufactured with different energy inputs but the same layer height and laser spot size. Whereas the energy input in the sample (A) was quite low and resulted in the formation of irregular porosities, the higher energy input in the sample (B) lead to the emergence of spherical porosities.

LPBF processed AlSi10Mg samples depending on the manufacturing direction. As can be seen in [Fig. 1.4](#), samples manufactured parallel to the tensile test direction showed an elongation of 4% while samples manufactured perpendicular to the test direction showed an elongation of about 8%, both with a UTS around 400 MPa. An additional challenge in the production of aluminum alloys in laser-based techniques operated in a protective gas atmosphere is the oxygen intake during the process,

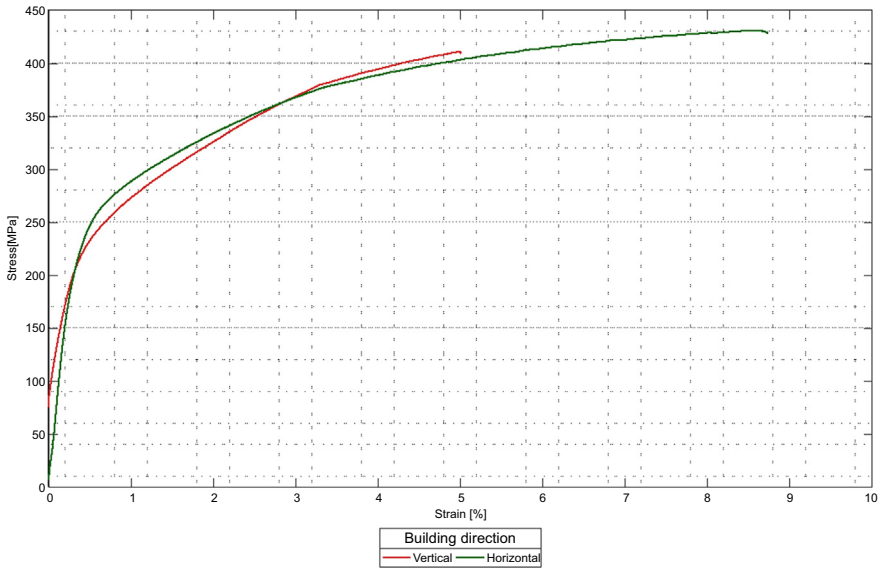


Figure 1.4 Tensile tests performed with samples manufactured out of AlSi10Mg by additive manufacturing. The samples fabricated with different building directions achieved almost the same Ultimate Tensile Strength. However, horizontally manufactured samples (building direction is perpendicular to tensile test direction) showed an almost doubled elongation when compared to the vertically manufactured samples (build direction is parallel to the tensile test direction).

which poses a challenge considering the recyclability of these alloys. Studies have shown that subsequent reuse of aluminum powders can decrease the properties of the material [14–16]. Additional difficulties arise when using laser-based manufactured parts due to the anisotropy of the printed material. Depending on the building orientation, among other things, due to columnar grain growth, mechanical properties of samples tested in parallel and perpendicularly to the build direction can vary greatly [17–19]. Studies at TU Graz have shown a difference in elongation in LPBF processed AlSi10Mg samples depending on the manufacturing direction. As can be seen in Fig. 1.4, samples manufactured parallel to the tensile test direction showed an elongation of 4% while samples manufactured perpendicularly to the test direction showed an elongation of about 8%, both with a UTS around 400 MPa.

1.1.2 Overview over aluminum alloys produced by additive manufacturing

The following section will provide an overview of selected aluminum alloys successfully produced by various additive manufacturing techniques and respective material properties, as well as special observations for each alloy.

1.1.2.1 *AlSi10Mg*

AlSi10Mg is the most commonly used material for additive manufacturing, whereas LPBF is the method most commonly applied. It is lightweight with a density of 2.68 g/cm^3 and has good materials properties, for example, a tensile strength of up to 450 MPa by an elongation of up to 8% and a hardness of up to 150 HV. This alloy has a chemical composition of Al-9.0–11.0Si-0.55Fe-0.20–0.45Mg-0.45Mn-0.15Ti-0.10Zn-0.05Cu-0.05Ni-0.05Pb-0.05Sn (wt.%). Due to its good weldability, it is perfectly suitable for additive manufacturing and the high cooling rates usually result in a fine grain structure [18–27].

The strengthening mechanism of this alloy is based on Si-rich precipitates that also act as nucleation sites for the fine grain structure, forming cellular structure in the as-printed parts [26]. Various heat treatments can result in a change in materials properties due to, for example, a change in the precipitate structure and grain coarsening in the as-printed parts. Some heat treatments may increase the density of the as-printed samples [28]. For instance, the most conventional heat treatments T4 (solution heat treatment), and T6 (artificial ageing) or annealing, lead to a decrease of strength and an increase of ductility. To perform T4 heat treatment, the samples have to be heated up to about 530°C , held there for a short time (2 h) and subsequently quenched with water [28]. To perform T6 heat treatment, the samples have to be treated with a T4 treatment at first and afterward, they are heated up again to about 155°C and held at this temperature for a longer time (12 h), followed by an air quench [28]. As studies at TU Graz have shown that the application of an annealing heat treatment at 300°C can lead to a reduction of the tensile strength almost by half while the elongation more than doubles, as demonstrated by Fig. 1.5. For the application of this type of heat treatment, the samples are heated up to 300°C and held there for 2 h, followed by a water quench. Also, regarding hardness, the trend is quite similar: The application of heat treatments like T6 usually leads to a decrease in the hardness of the printed parts. Heat treatments also influence the Charpy impact energy of this alloy, which can be enhanced from about 8 J, in the as-printed condition, to about 20 J as preliminary studies have shown (represented in Fig. 1.6). The magnitude of these changes depends on the temperature and the time of the corresponding heat treatment. Some treatments can even reduce the anisotropy of the printed parts [29].

Currently, studies about novel and adapted heat treatments especially for printed parts are made since the conventional heat treatments like T4 or T6 are not so well suited for additively manufactured aluminum alloys. Adapting prealloyed powders with pure elemental powders was also investigated. As Aversa et al. [30] found, the addition of pure Ni powder to prealloyed AlSi10Mg can lead to a great increase in the hardness of the fabricated samples. This increase of hardness is based on the formation of Al_3Ni precipitates. Moreover, Ni was well dispersed in the matrix.

1.1.2.2 *AlSi12*

AlSi12 is, besides AlSi10Mg, the second most used material for additive manufacturing, in reason of its great castability and low shrinkage [31–34]. AlSi12

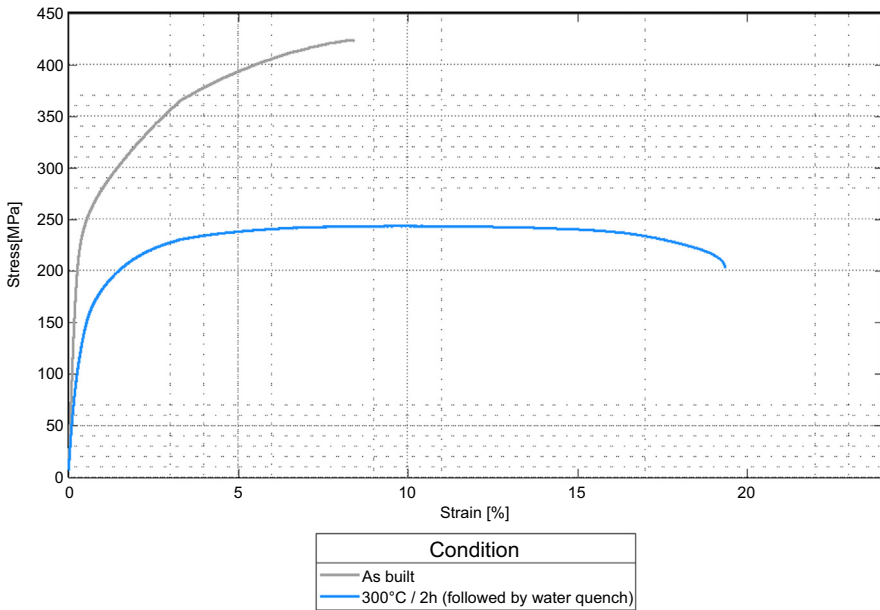


Figure 1.5 Tensile tests performed with samples manufactured out of AlSi10Mg by additive manufacturing. The annealing heat treatment (Samples are heated up to 300°C and held at this temperature for 2 h, followed by a water quench.) leads to a reduction of tensile strength almost by half, whilst the elongation more than doubles.

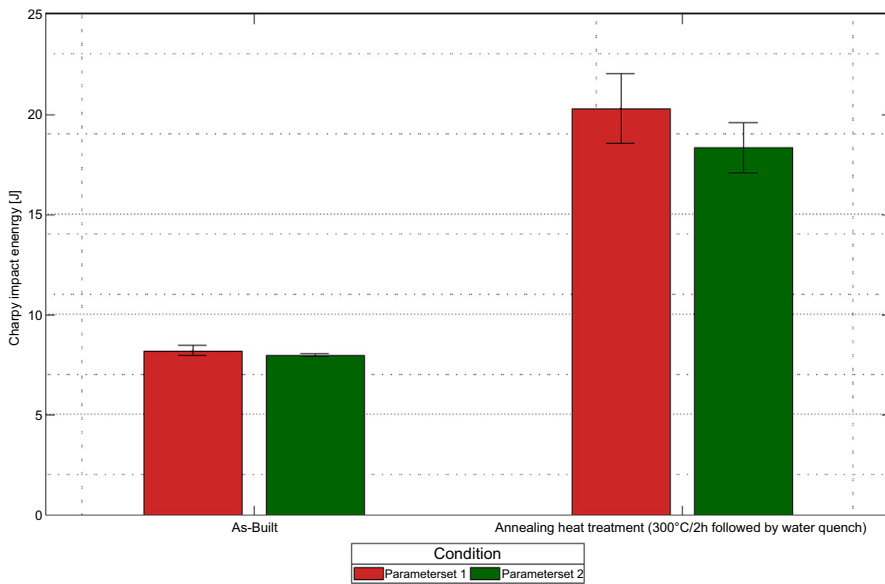


Figure 1.6 Charpy impact tests performed with samples manufactured out of AlSi10Mg by additive manufacturing with different sets of process parameters. The annealing heat treatment (Samples are heated up to 300°C and held at this temperature for 2 h, followed by a water quench.) leads to an increase of the Charpy impact for both processing conditions.

has a density of 2.66 g/cm^3 besides good mechanical properties. The chemical composition of AlSi12(Fe)(a) is $\text{Al-10.5-13.5Si-0.45-0.90Fe-0.55Mn-0.15Zn-0.15Ti-0.08Cu (wt.\%)}$. For instance, a tensile strength of up to 460 MPa by a quite poor elongation of up to 4%. The hardness reaches values up to 115HV in the as-printed condition [33]. Heat treatments do have big effects on the mechanical properties also for this alloy. Annealing, for example, results in a great decrease in strength, but also in an increase in ductility (up to 15%).

1.1.2.3 *Sc, Zr-based aluminum alloys*

Sc and Zr-based aluminum alloys are considered quite promising elements in the fields of additive manufacturing. These alloys are lightweight and can be strengthened by the precipitation of $\text{Al}_3(\text{Sc,Zr})$ particles during subsequent heat treatment. Considering several studies, the temperature of the heat treatment should not exceed 400°C due to the growth of the $\text{Al}_3(\text{Sc,Zr})$ precipitates and the related loss of coherency [35]. In the case of these alloys, most works were done with an alloy called ScalmaalloyRP, with a density of 2.7 g/cm^3 and a nominal composition of $\text{Al-4.6Mg-0.66Sc-0.42Zr-0.49Mn (wt.\%)}$. A tensile strength exceeding 500 MPa by an elongation of 14% in the heat-treated condition is achieved. The microhardness is also increased during annealing, which then reaches values up to 177HV [36–39]. Another promising alloy of this kind is named AA5024, with a density of 2.67 g/cm^3 and an average composition of $\text{Al-4.2Mg-0.4Sc-0.2Zr-0.18Mn-0.04Si-0.01Fe (wt.\%)}$. Studies have shown that an increase of hardness can be achieved even after a short annealing time at moderate temperatures [40–42].

As aforementioned, these kinds of alloys have a great susceptibility to cracking during the manufacturing process due to the high cooling rates within the process, which makes their processing challenging. It has been shown that Sc in these alloys can have a positive influence on the prevention of cracks as well as on the grain structure. As a grain refiner, Sc leads to smaller, equiaxed grains in comparison to the columnar grain structure in comparable alloys without Scandium by precipitating in Al_3Sc particles and acting as a nucleation site. In combination with Sc addition, higher energy inputs and a preheating can help form a more equiaxed and coarse grain structure, preventing an anisotropy in the sample as can be seen in Fig. 1.7 [41].

1.1.2.4 *Al-Cu alloys*

Al-Cu alloys belong to the wrought alloys and are considered hard to be manufactured by AM due to their low weldability. Fully dense parts can be obtained by LPBF, although no correlation between energy input and relative density can be found. Support structures, on the other hand, seem to influence the density of the printed parts, with samples printed on support structures having higher densities. This effect was attributed to the conservation of heat with the powder bed in between the support structures, this acting as an insulator for heat dissipation thus leading to slower cooling rates in the sample [43]. For the additive manufacturing

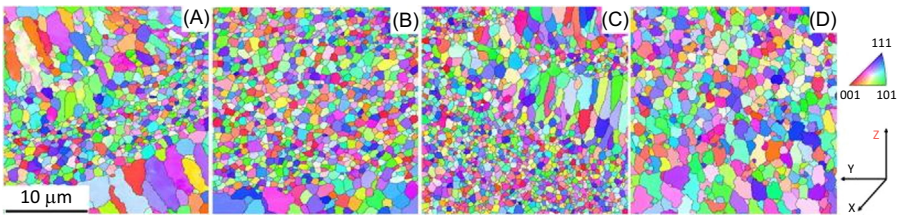


Figure 1.7 Microstructures reconstructed from inverse pole figures obtained from electron backscattered diffraction measurement of samples printed under different preheating conditions and with different printing parameter: (A) $E = 77.1 \text{ J/mm}^3$, platform temperature of 35°C ; (B) $E = 154.2 \text{ J/mm}^3$, platform temperature of 35°C ; (C) $E = 77.1 \text{ J/mm}^3$, platform temperature of 200°C ; (D) $E = 154.2 \text{ J/mm}^3$, platform temperature of 200°C . Higher platform temperature and higher energy inputs (E) lead to a more equiaxed, isotropic grain structure. *Source:* From K.V. Yang, Y. Shi, F. Palm, X. Wu, P. Rometsch, Columnar to equiaxed transition in Al-Mg(-Sc)-Zr alloys produced by selective laser melting, *Scr. Mater.* 145 (2018) 113–117.

of Al-Cu-Mg, it has been shown that dense parts ($> 99.7\%$) can be obtained with direct energy deposition (DED). The densification process here seems to be dependent on the deposition pattern, where bidirectional deposition resulted in a denser part than unidirectional deposition [44]. DED manufactured Al-Cu-Mg parts were mainly composed of α -Al and CuAl_2 , and a $[100]$ texture was observed in the building direction. Due to a heat treatment by deposition, grain coarsening in the middle part of the sample occurred. Ultimate tensile strength of 276 MPa with an elongation of 18% was achieved. Microhardness varied from 90HV (bottom part) to 135HV (middle part), which was explained with a heterogeneous microstructure distribution.

1.1.2.5 AA7075 alloys

The AA7075 alloy, with a norm composition of Al-5.1–6.1Zn-2.1–2.9Mg-1.2–2.0Cu-0.5Fe-0.4Si-0.3Mn-0.2Ti-0.18–0.28Cr (wt.%), is currently one of the strongest commercially available and is therefore used in the automotive, aviation, and construction industry. AA7075 is a heat-treatable alloy with the precipitation of Al_2CuMg (S-phase), $\text{Al}_2\text{Mg}_3\text{Zn}_3$ (T-phase) and MgZn_2 (η -phase). Studies on the additive manufacturing of AA7075 with LPBF have been done, but the mechanical properties are still much inferior to that of conventionally produced alloys due to a tendency of crack formation in this alloy [4]. Crack formation occurs due to the high cooling rates in the process, and heating of the building plates has shown to be not helpful in this regard [5]. However, crack formation may be prevented by the addition of Si to the powder; additions of up to 4% Si has been shown beneficial to prevent heat cracking [45]. Hardness values up to 171HV and yield strengths up to 279 MPa (as-built) and 338 MPa (heat-treated) in compression tests can be achieved with this method. Conversely, mechanical properties are still inferior to that of conventionally produced AA7075 alloys.

1.1.2.6 AA6061 alloys

Equally to AA7075, AA6061 alloy is prone to heat cracking during the LPBF process due to a tendency to form long-dendritic grains in the solidification process, leading to areas of molten metal between the dendritic grains that tend to rupture upon further solidification. It has a nominal composition of Al-0.8–1.2Mg-0.4–0.8Si-0.7Fe-0.15–0.40Cu-0.04–0.35Cr-0.25Zn-0.15Ti-0.15Mn (wt.%). The formed cracks then propagate upward upon deposition of further layers in the LPBF process. Numerical simulations and melt track studies have been done to understand the influence of the printing parameter on the solidification process in this alloy [6,7]. The heating of the powder bed up to 500°C in LPBF has been shown to improve the quality of the printed parts so that crack-free AA6061 can be produced. Mechanical properties similar to that of wrought AA6061 can be achieved by this method [8]. Additions of additives to the powder mixture has also been demonstrated to improve the quality of the printed parts in preventing crack formation. In a recent study, Martin et al. [10] demonstrated that the addition of hydrogen stabilized Zr nanoparticles to both AA6061 and AA7075 can prevent crack formation by acting as a nucleation site to produce a fine, equiaxed grain structure contrary to the commonly observed columnar structure. Additionally, Al₃Zr precipitations formed in the process act as a strengthening mechanism in both alloys.

1.2 Laser powder bed fusion of tool steels

Metal Additive Manufacturing (MAM), especially Selective Laser Melting (SLM), also known as Laser Powder Bed Fusion (LPBF), provides geometric design freedom for manufacturing metal parts. AM technique makes possible the fabrication of complex shapes for the tooling industry. In recent years, the use of steel for LPBF has been demanded by the tool and die-making industry. Molds and dies with conformal cooling channels are of great interest. Important goals are a reduction of cooling time by integrating conformal cooling strategies and an increase in tool life. For injection-molding technologies, LPBF-fabricated tools or inserts can achieve high productivity. Cooling is the most time-consuming step during injection molding of parts. In this case, cooling is the key factor [46].

1.2.1 Hot work tool steels

Maraging steels are widely used in additive manufacturing, especially LPBF [47]. A well-known representative of maraging steel is the grade 18Ni-300 with the chemical composition in wt.%, Fe bal., C ≤ 0.03, Ni 18.0–19.0, Co 8.5–9.5, Mo 4.7–5.2, Ti 0.5–0.8 according to the ASTM-standard [48]. This grade is almost carbon-free, with less than 0.03 wt.%. Maraging steel grades contain Ni and other alloying elements such as Co, Ti, and Mo, which act as hardening elements, thereby compensating the lack of hardening due to the very low carbon level. A low level of C leads to excellent welding and toughness properties. These steel grades reach

high strength and ductility [49]. In as-built condition, the 18Ni-300 microstructure contains martensite and retained austenite [50]. It is characteristic for LPBF of steel alloys and other metals, as an example 316 L (in wt.%, Fe bal., Cr 16.5–18.5, Ni 10.0–13.0, Mo 2.0–2.5, Mn $\leq$ 2, Si $\leq$ 1.0, C $\leq$ 0.03) [51] and AlSi10Mg (in wt.%, Al bal., Si 9.0–11.0, Mg 0.2–0.45, Mn $\leq$ 0.45, Fe $\leq$ 0.55) [52], that small melt pools lead to high cooling rates and a very fine cellular/dendritic microstructure is formed in as-built condition [50,53]. In the as-built condition of 18Ni-300, the martensitic phase is observed inside the cells. The retained austenite can be observed along the cell boundaries. Due to the microsegregation of alloying elements during solidification, the retained austenite remains in the intercellular region. These segregated zones are enriched with Ti, Ni, and Mo [54]. Ageing leads to a soft martensitic microstructure with high strengths caused by precipitation of intermetallic phase. High strengths are achieved by intermetallic precipitates of Ni₃(Mo,Ti) and Fe₂Mo during ageing [55,56]. Bodz et al. [55] reported that no precipitations can be found in as-built condition for the 18Ni-300 alloy. After ageing at 510°C for 2 hours, different phases such as spherical and plate-like precipitates enriched in Ti and Ni and a further spherical precipitate enriched in Mo are identifiable. The size of these precipitates ranged from 5 to 9 nm and the length of the plate-like precipitates ranged from 9 to 22 nm. The mechanical properties are achieved by a combination of nanometer-sized intermetallic precipitates in a martensitic matrix [49]. Armilotta et al. [57] reported the successful fabrication of hybrid diecasting tools with integrated cooling channels fabricated by LPBF-technology. Their results show that conformal cooling channels improve the surface finish of parts and lead to a reduction of cycle time and a reduction of shrinkage porosity of zinc alloy castings. Stache et al. [58] published the series production of press hardening parts using LPBF-fabricated tool segments of the maraging steel grade 18Ni-300. In comparison, one segment of the press hardening tool was conventionally manufactured from X38CrMoV5–3 (in wt.%, Fe bal., C 0.35–0.40, Cr 4.8–5.2, Mo 2.7–3.2, Mn 0.3–0.5, Si 0.3–0.5) [59]. Direct comparison of the tool segments demonstrated the higher material removal (abrasion) of the additively manufactured insert. In this study, the processing of the hot work tool steel X38CrMoV5–3 by LPBF is also investigated. Huskic et al. [60] demonstrate the successful use of LPBF-fabricated 18Ni-300 forging dies under industrial conditions. The additively manufactured parts withstand the high thermomechanical loads during forging. In this study, it was clearly stated that the use of process-optimized hot work tool steels is necessary. To meet the material-specific requirements of hot, cold, and high-speed application, there has been a high interest in processing tool steels by LPBF in recent years.

The three groups of steels for hot forming applications are Cr-, W- and Mo-hot work tool steels [61]. Examples of conventional hot work tool steels are H11 (in wt.%, Fe bal., C 0.33–0.43, Cr 4.75–5.50, Mo 1.1–1.6, Mn 0.2–0.6, Si 0.8–1.25, V 0.3–0.6) [62], H13 (in wt.%, Fe bal., C 0.32–0.45, Cr 4.75–5.50, Mo 1.1–1.75, Mn 0.2–0.6, Si 0.8–1.25, V 0.8–1.2) [62], and W360 (in wt.%, Fe bal., C 0.50, Cr 4.50, Mo 3.00, Mn 0.25, Si 0.20, V 0.55) [63]. The requirements for these steel grades are high thermal conductivity, wear resistance, hardness, and sufficient

toughness. The C-alloyed tool steels contain carbide-forming elements such as Cr, V, Mo and W. Tool steels have excellent hardness, wear resistance, and good resistance to high temperature softening. Traditionally, a heat treatment leads to a martensitic matrix with fine distributed carbides, the latter responsible by improving wear resistance and hot hardness [61]. Table 1 shows the mechanical properties of LPBF-fabricated hot work tool steel grades. Hot work tool steels H11, H13, and W360 have a carbon content of 0.32–0.50 wt.%.

H13 is a widely used hot work steel. This tool steel grade is commonly used for forging dies, extrusion dies and die-casting. Published studies have focused on processing H13, a Cr-Mo-V martensitic hot work tool steel grade [68–71]. The main problems with processing these C-containing hot work tool steel grades are distortion, cracking and delamination [58,67–69,71,72]. Typical are long cracks from the surface to the center of the part (Fig. 1.8). In these studies, fabrication by LPBF depends on the optimization of process parameters to reach a high material density which is achieved by reducing defects such as pores, lack of fusion, unmelted powder particles, inclusions and cracks. Most of the current research considers the

Table 1.1 Overview and mechanical properties of hot work tool steels processed by LPBF (heat-treated).

AISI	DIN	Carbon [wt. %]	YS/UTS [MPa]	A [%]	Hardness [HRc]	Author
18Ni-300	1.2709	< 0.03	1977/1982	4.5 ± 0.4	56	Simon et al. [64]
H11	1.2343	0.33–0.43	-/2148	8.8 ± 1.1	54	Huber et al. [65]
H13	1.2344	0.32–0.45	1512/1894	10 ± 1	54	Oerlikon [66]
W360 AMPO	—	0.50	1680/2000	7	56	Giedenbacher et al. [67]
M789 AMPO	—	<0.02	1600/1800	7	52	Turk et al. [49]

YS, Yield strength; UTS, ultimate tensile strength; A, elongation at break; H, hardness.

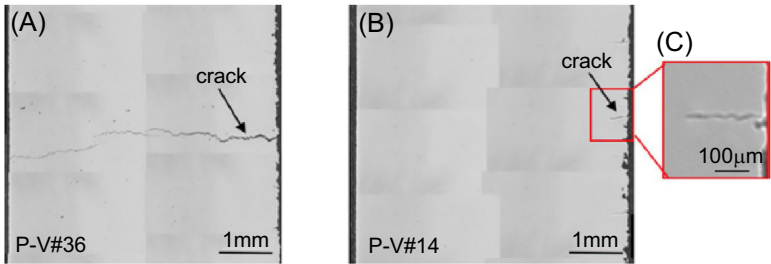


Figure 1.8 Typical crack formation of H13 hot work steel fabricated by LPBF.

Source: From Y. He, M. Zhong, J. Beuth, B. Webler, A study of microstructure and cracking behavior of H13 tool steel produced by laser powder bed fusion using single-tracks, multi-track pads, and 3D cubes, J. Mater. Process. Technol. 286 (2020) 116802.

volume energy density (VED) as the key factor for the properties of as-built parts. The VED is given by $VED = P/(v * h * a)$ [J/mm³], where P is laser power, v is laser-scanning speed [mm/s], h is powder layer thickness [mm] and a is hatch distance [mm]. VED describes a very simplified approach. It does not involve the absorption of the laser radiation, spot diameter, scanning strategy and the substrate preheating temperature [73,74].

Researchers have focused on the development of process windows for LPBF-densified martensitic tool steels [65,67–69,71]. During the cooling down of C-containing hot work tool steels, in solid-state, the austenite temperature decreases below martensite start temperature and thus the phase transformation takes place. Carbon strongly influences the formation of martensite. In this case, quenched and untempered martensite is brittle and contains a high level of residual stresses, leading to crack formation. The build-up of further layers by melting and solidification leads to intrinsic heat treatment (cyclic heating and cooling). As a result, an inhomogeneous microstructure leads to hardness differences. Mertens et al. [70] and Giedenbacher et al. [67] investigated the effect of the preheating temperature on the processing of hot work steel grades H13 and W360 by LPBF. These studies reported a more homogeneous microstructure at higher preheating temperatures. The brittle martensitic phase will be tempered layer by layer with the laser. At a higher preheating temperature a phase transformation and a change of microstructure can be observed. A fine bainitic microstructure with high hardness is observed at a preheating temperature range of 300°C–450°C. It is observed that preheating above martensite-start leads to an isothermal formation of bainite in as-built condition [65,67,70]. A high amount of retained austenite in the as-built condition is also reported. Giedenbacher et al. [67] found out that substrate preheating extends the process parameter window for W360 hot work tool steel. However, there are also adverse effects, such as increased oxidation of the powder particles and the solidified layers. High preheating temperatures of more than 500°C show a significant hardness decrease in as-built condition of C-alloyed hot work tool steel grade. The chemical analyses show increased decarburization (reduction of carbon) in LPBF-samples fabricated by high-temperature substrate preheating. In this case, decarburization of 20.8%–22.9% can be detected. Zhao et al. [75] report decarburization of 21% in specimens during the LPBF process of AISI 420 (in wt.%, Fe bal., C 0.39, Cr 13.9, Mn 0.73, Si 0.27, V 0.32) powder. Oxygen measurements have shown that an increase in preheating temperatures leads to a rise in the oxygen level in SLM specimens. This result is in agreement with observations of Krell et al. [69], who reported an increased level of oxygen after LPBF. It must be noted that substrate preheating leads to an increased oxygen level in the powder particles and LPBF specimens. A high purity, low oxygen process atmosphere is an essential influencing factor for fabricating high material quality. Krakhmalev et al. [76] show that lower layers contain a higher amount of retained austenite due to an in situ carbon partitioning effect during fabrication of martensitic stainless steel AISI 420. The characteristics of the LPBF process are high cooling rates and cyclic temperature changes, which affect the microstructure and the mechanical properties of parts. The LPBF-fabricated hot work tool steels also show a hierarchical microstructure.

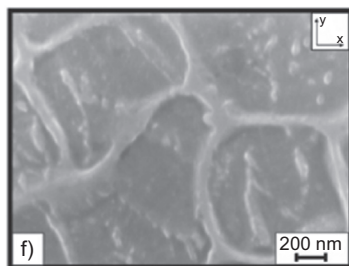


Figure 1.9 SEM-photograph of LPBF fabricated H13 specimen—cellular microstructure in as-built condition.

Source: From J. Krell, A. Röttger, K. Geenen, W. Theisen, General investigations on processing tool steel X40CrMoV5-1 with selective laser melting, *J. Mater. Process. Technol.* 255 (2018) 679–688.

Due to the high cooling rates, hot work tool steels consists of a very fine cellular/dendritic microstructure and nanoscale precipitation [67,69,70]. Krell et al. [69] reported no change in the microstructure up to 300°C substrate preheating during the processing of H13-tool steel by LPBF. The rapid solidification during LPBF leads to microsegregations. Etching with 3 mol% nital causes a darker phase of ferritic nature and a brighter austenitic phase. The ferrite cells have a size of 0.5–2 μm and a thickness of 0.1–0.2 μm . Small precipitations can also be detected inside the cells, as depicted in Fig. 1.9. As mentioned before, the prevention of cracking during LPBF of tool steel is a major challenge. Substrate preheating significantly reduces crack formation. Narv et al. [68] reported relative densities up to 99.9% of LPBF-fabricated H13 tool steels.

1.2.2 High-speed steels and cold work tool steels

In recent years, the processing of cold work and high-speed steels by LPBF has been of great interest. The crack-free processing of such high carbon alloyed wear-resistant steels is reported by Saewe et al. [77], Geenen et al. [78] and Kempen et al. [79]. In these studies, the high-speed steels HS 6–5–3–8 (in wt.%, Fe bal., C 1.33, Co 8.3, Cr 4.1, Mo 4.0, Mn 0.3, V 3.0, W 6.3, Si 0.5), M3:2 (in wt.%, Fe bal., C 1.29, Cr 3.90, Mo 4.8, V 3.0, W 6.2, Co 0.48) and M2 (in wt.%, Fe bal., C 0.9, Cr 3.97, Mo 4.89, Mn 0.38, V 1.82, W 6.15, Si 0.35) were successfully processed [78–80]. Boes et al. [80] and Platl et al. [81] reported the processing of cold work steels by LPBF. The properties of such steels are accompanied by high hardness with low ductility in as-built condition. As a result, the challenges are cracking and delamination which are mainly caused by residual stresses during LPBF. The stresses are distributed differently within a component's structure. Mercelis et al. [82] reported that the maximum compressive stress is reached at the surface and tensile stresses occur in the z-direction at the base plate. Substrate preheating leads to a reduction of the residual stress level by lowering the temperature gradient. This

mechanism is responsible for the effective reduction of residual stresses. As a consequence, the yield strength decreases with increasing temperature for many materials [70,82,83].

The primary structure of a welding lens in the above-mentioned high-speed steels and cold work tool steels appears as a finely dendritic, cellular or network-like structure in various forms. The microstructure consists of martensite, retained austenite and interdendritic carbides. The laser melting process leads to fine dendrites that are aligned in the direction of the heat flow [78–80,84,85]. The primary dendrite arm spacing is approximately $1\text{ }\mu\text{m}$ and secondary dendrite arm formation is prevented by the high cooling rate. Different substrate preheating temperatures show no effect on the primary dendrite spacing. Epitaxial growth is observed in both Sander et al. [85] and Saewe et al. [84] studies and is referred to as martensite plates that extend across dendrites and melt paths respectively. In Fig. 1.10, two scanning electron microscope images of polished transverse sections at a preheating temperature of $T_{H,1} = 200^\circ\text{C}$ and $T_{H,4} = 600^\circ\text{C}$ are shown for the HS 6–5–3–8. This fine cellular structure consists of network-like carbides and austenite, which in turn enclose coarsely structured areas of martensite plates. When the substrate plate temperature is increased, a growth of these plates can be observed, which break through the network-like structure above a certain size [80,85].

The different substrate preheating temperatures lead to altered cooling mechanisms which are reflected in the ratio of face-centered cubic to body-centered cubic crystal structures. As a result, depending on the alloy, the retained austenite content increases up to a temperature of 200°C and then decreases again. The increase of the martensitic phase at higher temperatures is based on a temperature-induced transformation of retained austenite into martensite [77,85]. The different substrate preheating temperatures lead to altered cooling mechanisms, which are reflected in the ratio of face-centered cubic to body-centered cubic crystal structures. As a

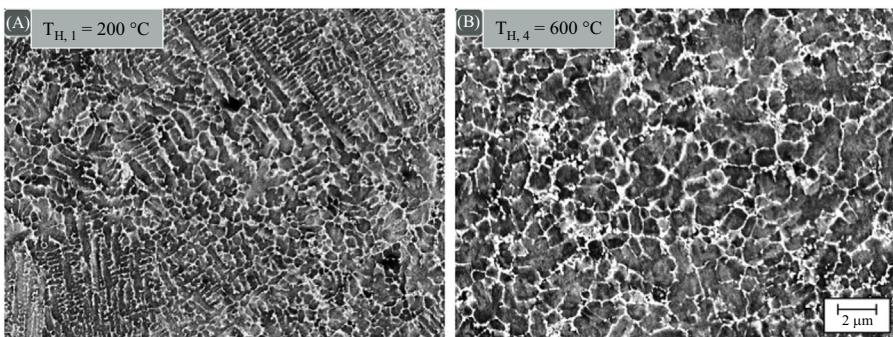


Figure 1.10 Scanning electron microscope images in secondary backscattered electrons mode of polished cross-sections of specimen built at preheating temperatures (A) $T_{H,1} = 200^\circ\text{C}$ and (B) $T_{H,4} = 600^\circ\text{C}$ [77].

Source: From J. Saewe, M.B. Wilms, Influence of preheating temperature on hardness and microstructure of PBF steel HS6-5-3-8. (2019).

result, depending on the alloy, the retained austenite content increases up to a temperature of 200°C and then decreases again. The increase of the martensitic phase at higher temperatures is based on a temperature-induced transformation of retained austenite into martensite [80,85].

1.3 Laser metal deposition of steels

1.3.1 Introduction

In Laser Metal Deposition (LMD), also known as Laser Cladding or Direct Energy Deposition, a laser creates a molten pool on a substrate. A powder is conveyed in an inert gas stream through the powder nozzle and introduced into the molten pool. Gradually, layer-wise deposited weld beads form a structure on existing base bodies or entire components. This process can be combined, for example, with a 5-axis milling machine, thus combining the high precision and surface quality of milling with the high flexibility, freedom of design, and build-up speed of laser build-up welding. In contrast to bed-based powder processes, the material is only applied where it is needed and no support geometries are required. The flexible alternation between laser and milling processing enables the finishing of component segments on the finished part that cannot otherwise be reached due to the component geometry. This hybrid process can be used to manufacture and repair tools and molds or to apply wear-resistant coatings to stressed component surfaces.

Laser Additive Manufacturing in the field of Deposition Welding has benefited from further development in the field of special CAM systems. For example, the adaptive tool-path deposition method was developed for the manufacture and repair of turbine blades [86,87]. Compared to other repair processes such as Tungsten Inert Gas welding (TIG), the heat input or distortion and the change in metallurgical properties of the substrate are relatively low. For this reason, the LMD process is well suited for the repair of tools [88].

The steels used for tools are usually hardenable and therefore have a relatively high carbon content. During the processing of these materials, defects can occur in the form of cold and hot cracks which subsequently lead to the failure of the component [88]. The processing of tool steels by LMD is a current research area of various publications.

1.3.2 LMD of tool steels

Tool steels are used in different areas and can be divided into hot work, cold work, and high-speed steels. Hot work steels are mainly used in hot forming manufacturing processes where high thermal and mechanical loads occur. The properties of the steels are given by their chemical composition, processing methods, and heat treatment and influence productivity and thus production costs and component quality.

Tools made of hot-work tool steels are usually exposed to high mechanical and thermal stresses and thus have a limited service life. For this reason, properties such as high resistance to deformation and cracking due to mechanical and thermal stresses are required in the area of hot forming, which are mainly influenced by the tool manufacturing process [89]. The tooling costs account for about 10% of the production costs in, for example, forging [90]. Of the 10% of tool costs, 46% can be attributed to the wear of the forging dies [91]. This initial situation is driving research activities in the field of innovative manufacturing processes in toolmaking.

Bohlen et al. [92] investigated the processability of tool steel DIN 1.2344 (Fe-5.40Cr-1.35Mo-1.00V-1.00Si-0.39C wt.%) using LMD. The processability and the resulting microstructure of the material were determined based on thin walls. Due to the layered structure during additive manufacturing plus the resulting repeated heating and quenching cycles, heat treatment occurred during the manufacturing process. In the case of hot-work tool steel 1.2344, this heat treatment has a significant influence on the resulting microstructure. Due to the high cooling rates, the temperature falls under the martensite starting temperature before reheating; as a result, martensite is formed. Subsequent reheating causes reaustenitization of the martensite. Martensitic structures that are not reaustenitized due to the insufficient temperature are tempered by the repeated heat input. This martensite formation, reaustenitization, and tempering effects are highly dependent on the geometry, process parameters, and position within the parts. In the last layer of the part, reheating does not occur. The result is nontempered martensite and thus high hardness values on the sample surfaces while the tempered areas have lower hardness values, resulting in an inhomogeneous microstructure within a component. Accordingly, the built-up structures exhibit inhomogeneous properties due to the different heat inputs. Therefore, according to the authors, subsequent heat treatments are necessary to achieve homogeneous postprinting properties.

Junker et al. [93] determined a process window for the tool steel DIN 1.2343 (Fe-5.15Cr-1.3Mo-0.40V-1.00Si-0.37C-0.38Mn wt.%) for the repair of forming tools using LMD by investigating the influence of different heat treatment strategies on the material properties. For this purpose, cuboids were built up from which specimens were taken for compression and tensile tests. Five specimens each ($n = 5$) were tested in as-built, hardened, heat-treated (hardened and tempered), and only tempered conditions were subsequently compared with conventionally produced material. Fig. 1.11 shows the hardness of the samples tested. In the as-built condition, an increase in hardness of the soft annealed substrate from 300 HV_{0.5} to 730 HV_{0.5} in the additively manufactured part was measured. The additively manufactured part in the as-built condition exhibits hardness values similar to those in the hardened condition. After hardening, the substrate also exhibits similar values. Concerning tool repairs, it would be advantageous to be able to dispense with conventional heat treatment (hardening and tempering). In the LMD process, the welded material is hardened due to rapid cooling and the hardening step can theoretically be skipped in subsequent heat treatment. For this reason, samples are manufactured in which only a threefold tempering process after the LMD process is carried out. These specimens also showed similar hardness values to the hardened samples.

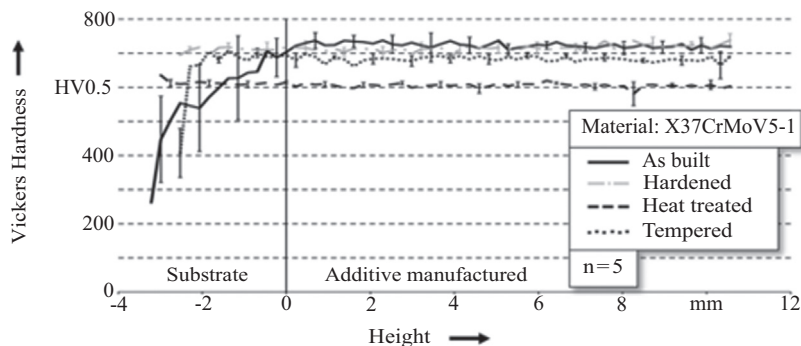


Figure 1.11 Hardness in building direction.

Source: From D. Junker, O. Hentschel, M. Schmidt, M. Merklein, Investigation of heat treatment strategies for additively-manufactured tools of X37CrMoV5-1, *Metals (Basel)*. 8 (2018) 1–13.

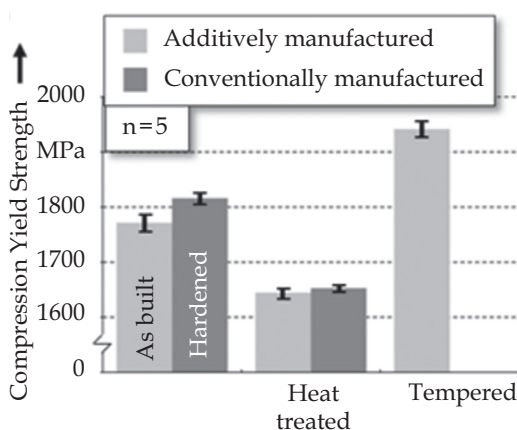


Figure 1.12 Compression Tests, compression yield strength.

Source: From D. Junker, O. Hentschel, M. Schmidt, M. Merklein, Investigation of heat treatment strategies for additively-manufactured tools of X37CrMoV5-1, *Metals (Basel)*. 8 (2018) 1–13.

A compression test showed that the specimens ($n = 5$ for each heat treatment strategy) tempered three times had the highest strengths (Fig. 1.12). Conventional heat treatment resulted in a decrease, only hardening in a slight increase in strength.

In a tensile test depicted in Fig. 1.13, the specimens ($n = 5$ for each heat treatment strategy) tempered three times also showed the highest strengths. In this case, conventional heat treatment also led to a decrease in strength (Fig. 1.3A). A comparison with conventionally manufactured material was made and it was found that the additively manufactured structures exhibit similar strengths after conventional heat treatment. If austenitization and hardening are dispensed with and the material is only tempered three times, higher strengths may be achieved. The elongation at

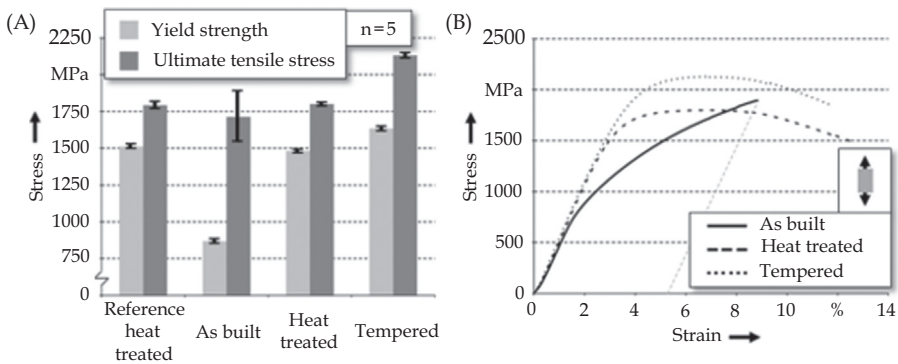


Figure 1.13 Tensile test, yield strength, and stress-strain curves in different conditions.

Source: From D. Junker, O. Hentschel, M. Schmidt, M. Merklein, Investigation of heat treatment strategies for additively-manufactured tools of X37CrMoV5-1, Metals (Basel). 8 (2018) 1–13.

break (Fig. 1.3B) determined in the tensile test is lowest in the as-built condition. The highest elongation at break can be achieved in the conventionally heat-treated condition. The specimens tempered only three times are slightly less elongated than the heat-treated ones at higher yield strength and ultimate tensile strength [93].

Metallographic examination revealed a comparatively fine dendritic solidified microstructure with retained austenite in the as-built condition. In addition, areas in which an increased proportion of chromium and molybdenum is present were found. It is assumed that the carbide content is higher there. During austenitizing and subsequent hardening, the structure in the as-built condition is dissolved and a homogeneous martensitic structure is present as in conventionally produced material. The carbide-forming elements are distributed homogeneously after heat treatment. If conventional hardening is not carried out and the material is only tempered three times after the LMD process, the low temperatures lead to a transformation of retained austenite to martensite. The fine-grained structure and the accumulation of chromium and molybdenum from the LMD process are retained. It is assumed, that the finer microstructure leads to higher strength in compression and tensile tests compared to heat-treated samples where the microstructure is completely dissolved. The accumulation of carbides could lead to a higher hardness, which is the reason for a relatively high compression strength [93].

Rabiey et al. [88] also conducted a study on the processability of the DIN 1.2343 for applicability in the field of tool repair. It was pointed out that weld beads show no porosity, and good bonding to the substrate was achieved. Due to inhomogeneous hardness values in the as-built condition, which were attributed to differences in the microstructure, it was assumed that heat treatment of the tools repaired by LMD is necessary (Fig. 1.14).

Telasang et al. [95] investigated the influence of different process parameters on the microstructure and hardness when tempered DIN 1.2343 tool steel bulk material was coated by LMD with one layer of the same material in powder. The

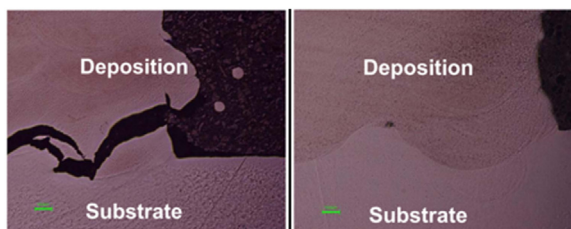


Figure 1.14 Cross-section of a multilayer deposited sample without (left) and with (right) preheating up to 600°C [94].

Source: From D.S. Shim, G.Y. Baek, E.M. Lee, Effect of substrate preheating by induction heater on direct energy deposition of AISI M4 powder, *Mater. Sci. Eng. A* 682 (2017) 550–562.

cross-section revealed a microstructure consisting of martensite, retained austenite, and fine carbides. The distribution of the individual phases changed with distance from the surface. The specimens built up by LMD are divided into the coated zone, the interdiffusion zone, the heat-affected zone, and the substrate exhibiting different hardness values in the as-built state. In the coated area (average 620 HV_{0.2}), a predominantly dendritic solidified microstructure with finely distributed carbides—alloy carbides in different stoichiometric compositions—in the interdendritic areas were found. In the interdiffusion zone (average 640 HV_{0.2}) mainly columnar dendrites were found. The heat-affected zone can be divided into two areas. At first, the area directly adjacent to the interdiffusion zone consisting of plate-like and acicular martensite does not melt during the welding process with an average hardness of 670 HV_{0.2}. By increasing distance to the coating, high-tempered martensite was found. This area, also not melted, was heated above the secondary hardness temperature. It led to a decrease in this property to an average of 430 HV_{0.2}, being softer than the substrate (average hardness of 490 HV_{0.2}). By using a pulsed laser, the described microstructure could be refined. The phase composition remained the same. Subsequent heat treatment with the laser led to further carbide precipitation by tempering the martensite or transformation of the retained austenite to pearlite and bainite.

Vollmer et al. [96] used the LMD process to partially apply a wear-resistant coating on critical areas of a forming tool made of DIN 1.2343 tool steel. Radii were coated with nickel-based powder alloys (Table 1.2). To evaluate the bonding strength of the different coatings a specially developed testing device was used [97]. The combination of Ni25 and 1.2343 showed the best results with an ultimate tensile force of 9471 N, while Ni40 and NiBSi reached only a quarter of the bonding strength. Samples made with Ni50 were too brittle for testing the bonding strength. To enhance wear resistance, the top layers of the Ni25 samples that showed the best results regarding defects were reinforced with tungsten carbides embedded in the last Ni25 layer. Light microscopic investigations of these samples showed only uncritical pores. Subsequent milling operations of the reinforced surfaces demonstrated significant wear of the cutting tool.

Table 1.2 Chemical composition of coating materials.

Material	Ni	Cr	C	Fe	Si	B
Ni25	bal.		<0.5	<1.00	2.50	1.70
Ni40	bal.	6.00	0.25	2.00	3.50	1.30
Ni50	bal.	11.00	0.45	2.50	3.80	2.30
NiBSi	bal.		0.03	0.40	3.00	3.00

Muro et al. [98] investigated the workability of a nickel-alloyed DIN 1.2343 tool steel. By adding one to three per cent nickel, the residual austenite content at room temperature was increased; in contrast, the hardness was reduced. Subsequent machining of the components coated with this material can thus be carried out in the soft state. Next, the samples were subjected to cryogenic treatment and cooled down to -175°C . In this process, the residual austenite transformed into martensite and a significant increase in hardness were measured. Tools coated by LMD usually have to be finished by milling. When using hot work tool steels with high carbon content, high hardness values due to the high cooling rates are common, becoming disadvantageous during machining. With the introduced manufacturing method, this drawback can be reduced.

High hardness, strength, and resistance to tempering at high temperatures characterize high-speed steel. The properties depend mainly on the type, morphology, volume fraction, and distribution of carbides. These are influenced by the cooling conditions during production. In conventional production by casting and forging, the slow cooling of 10–100 K/s during casting results in a comparatively coarse microstructure and segregation of carbides [92,99,100]. Cooling rates of up to $10^3\text{--}10^4$ K/s are achieved with the LMD process [101]. These high cooling rates lead to a fine microstructure, which in turn has a positive effect on the material properties [102]. The high cooling rates can lead to high stresses and subsequent cracks, especially in these materials.

Rahman et al. [103] investigated the processing of vanadium-rich high-speed steel DIN 1.2343 (Fe-9.0 to 12.0V-5.2 Cr-1.8 to 2.2C-1.2Mo-1.00Si wt.%) and characterized the microstructure and wear resistance as a function of different welding speeds using LMD. Microhardness and carbide size and shape are directly related to the welding speed and cooling conditions. A high welding speed leads to higher hardness and angular, rod-shaped carbides. Lowering the welding speed results in lower hardness, rounder carbides, and also a lower wear rate. These LMD-manufactured surfaces are compared to cast tools. The microstructure shows a very fine structure compared to the casting process. Furthermore, it is stated that preheating is necessary to avoid cracks. Samples produced at room temperature showed macro cracks.

Shim et al. [94] investigated the influence of different preheating temperatures on the material properties of the high-speed steel DIN 1.3351 (Fe5.88W-4.88Mo-4.25Cr-4.12V-1.33C-0.33Si-0.3Ni-0.26Mn-0.25Cu wt.%) during processing by LMD. Fig. 1.14 shows a cross-section of a multilayer coating produced with and

without preheating. Preheating and the resulting reduced cooling rate leads to the fabrication of crack-free specimens. Without preheating, the thermal stresses caused by the temperature difference between the molten bath, the substrate material, and the resulting rapid solidification of the molten material lead to cracks in the material. The preheating of the substrate also has a significant effect on the microstructure. By influencing the cooling rate, the solidification speed is also changed. In addition to a coarser structure, larger distances between the secondary dendrite arms were found. Without preheating, the main part of the microstructure consisted of equiaxial small grains. Preheating resulted in columnar dendritic growth. The changes were attributed to the lower cooling rate and the resulting slower solidification. The hardness of the additively produced specimens with and without preheating does not differ significantly, reaching up to 860 HV_{0.2}. The high hardness in the preheated specimens was attributed to possible carbide precipitation caused by the high preheating temperatures. In the tensile test, a very brittle fracture behavior was determined. The preheated specimens reached a maximum tensile strength of 950 MPa with and 820 MPa without preheating. The brittleness was attributed to the high hardness of the untempered martensite and the precipitates, which led to low ductility and toughness. During the fracture surface investigation, it was found that the fracture partially originates from pores. In the additively manufactured part of the samples, there was an intergranular fracture related to local microvoid coalescence. In the area between the substrate and the additively manufactured structure, an interdendritic fracture was found and attributed to carbide precipitation at the dendrite boundaries.

Park et al. [104] investigated the influence of energy density on the material properties of coldwork steel 1.2379 during LMD processing. It was found that hardness in the as-built condition decreases with increasing energy density. On the one hand, this is attributed to the changed cooling conditions and the slower solidification. With higher energy density, a coarser microstructure or larger distances between the dendrite arms are measured. On the other hand, increasing energy density is accompanied by a decrease in carbon content. During the LMD process, the carbon reacts with oxygen from the environment and with the metal powder itself. CO gas is formed which is outgassed and leads to a decrease in the carbon content in the material and subsequently to a decrease of the hardness.

1.3.3 Conclusion

When processing tool steel powder by LMD, specific challenges arise depending on the alloy. During production, the powder melts cool down and solidifies, and during cooling, a microstructural transformation from austenite to martensite takes place. These high cooling rates compared to conventional processes such as casting, create stresses that can lead to cracks. Depending on the alloy, preheating is necessary. Depending on the process parameters, material properties are inhomogeneous. Subsequent heat treatment of the built-up structures is necessary for homogenization. In addition to the above-mentioned challenges, the LMD process offers new possibilities, especially in tool-making and tool repair, and is, therefore, a promising

resource-saving manufacturing technology that can also save production steps and time [88,105,106]. Since the 1990s and until nowadays, remarkable developments have been done on research and application of SMAs, resulting in innovative products. Most of these innovations are based on the functionalities of such class of materials—namely shape memory effect and superelasticity—and due to that considerable possibilities for functional integration are possible [107]. For this reason, SMAs has been included in “smart” systems with adaptive and/or “intelligent” functions, such as sensors, actuators, and microcontrollers. Even though SMAs has been successfully established, manufacturing and processing are still challenging. Regarding NiTi, it is conventionally produced through casting. However, even under vacuum an increase in the impurity level (e.g., carbon and oxygen) degrade the functional properties, as secondary deleterious phases form during the solidification. An additional obstacle is related to machining, as it is used to form the final shape of the part. Excessive tool wear and spring back effect constitute hindrance on the subtractive step, whereas geometrical limitations restrict the applications. Nevertheless, additive manufacturing (AM) has proven to be a viable solution to most of the aforementioned problems, becoming a recurrent choice for SMAs component fabrication [108,109]. Based on the aforementioned, one intends to relate recent advances on the powder-based additive manufacturing of shape memory alloys, considering the manufacturing processes, classes of SMA, inherent metallurgical phenomena related to the fabrication, and its impact on the functional properties.

1.4 Powder-based additive manufacturing of shape memory alloys

1.4.1 Introduction

Since the 1990s and until nowadays, remarkable developments have been done on research and application of SMAs, resulting in innovative products. Most of these innovations are based on the functionalities of such class of materials—namely shape memory effect and superelasticity—and due to that considerable possibilities for functional integration are possible [107]. For this reason, SMAs has been included in “smart” systems with adaptive and/or “intelligent” functions, such as sensors, actuators, and microcontrollers. Even though SMAs has been successfully established, manufacturing, and processing are still challenging. Regarding NiTi, it is conventionally produced through casting. However, even under vacuum an increase in the impurity level (e.g., carbon and oxygen) degrade the functional properties, as secondary deleterious phases form during the solidification. An additional obstacle is related to machining, as it is used to form the final shape of the part. Excessive tool wear and spring back effect constitute hindrance on the subtractive step, whereas geometrical limitations restrict the applications. Nevertheless, additive manufacturing (AM) has proven to be a viable solution to most of the

aforementioned problems, becoming a recurrent choice for SMAs component fabrication [108,109]. Based on the aforementioned, one intends to relate recent advances on the powder-based additive manufacturing of shape memory alloys, considering the manufacturing processes, classes of SMA, inherent metallurgical phenomena related to the fabrication, and its impact on the functional properties.

1.4.2 Powder-based additive manufacturing of shape memory alloys: current technologies in use

Table 1.3 lists works performed on the powder-based AM of SMAs in different categories. According to recent reviews [108,109,117,152,153], Selective Laser Melting (SLM) and Selective Laser Sintering (SLS) are notably the most employed powder bed techniques. Electron Beam Melting (EBM) has also a potential for fabricating NiTi parts, even more, due to the vacuum chamber necessary for a proper equipment operation, thus reducing the impurity pickup. Also, direct energy deposition (DED) processes are employed, being laser engineered net shaping (LENS) widely used as a manufacturing way. Hence, these reviews in AM of SMA have focused their information on, e.g., optimum process parameters, build-up residual stresses, postprocessing treatments, microstructural and phase transformation behaviors, and the effect of defects on the mechanical properties [108,109,118,154,155]. As a matter of exploring how the shape memory materials are affected by the manufacturing techniques listed in Table 1.3, a brief analysis of the most recent works regarding the influence of additive manufacturing on printability, microstructure, transformation temperatures, and functional properties is put forward.

It is widely accepted the central role of the process parameters on the resulting microstructure and corresponding chemical composition of AM samples. Each set of parameters creates complex thermal profiles throughout the material during the melting-solidification-remelting cycles, fostering successive heat-affected zones (HAZs) and nonequilibrium solidification conditions in the melt pools. It promotes complexity on both phase transformations and precipitation mechanisms, resulting in a multitude of possible microstructures that affects directly mechanical and

Table 1.3 Processes used on the additive manufacturing of SMAs.

Category	Process	References	Feedstock
Powder bed fusion	Electron beam melting (EBM)	[110–113]	Metal powder
	Selective laser sintering (SLS)	[114–116]	
	Selective laser melting (SLM)	[117–137]	
Direct energy deposition	Laser metal deposition (LMD)	[138–146]	Metal powder or metal wire
	Laser engineered net shaping (LENS)		
	Plasma arc deposition (PAD)		
Binder jetting	Powder bed and inkjet 3D printing (3DP)	[147–151]	Metal powder

physical properties [156]. These facts have led numerous studies to assess the influence of laser power, scanning speed, hatch distance, scan track, and powder layer thickness on porosity, elemental evaporation, intermetallic precipitation, texture, and resulting transformation temperature of powder bed fusion additively manufactured shape memory alloys [117,119,157–159].

Wang et al. [157] applied a wide variation of process parameters on the SLM of NiTi, namely scanning speed from 400 to 1200 mm/s, hatch spacing from 40 to 110 μm , and laser power from 60 to 200 W, obtaining the DSC results depicted in Fig. 1.15 for describing the transformation behavior of the SLMed samples. Here, each pair of curves represent the samples in as-built (A, C, E) and solution treated (1000°C for 2 h) (B, D, F) conditions, and how variations in scanning speed (A, B), hatch spacing (C, D), and laser power (E, F), affect the transformation temperatures and peak shape thus enthalpy of transformation, that is, how spontaneous the reaction is. Their findings may be summarized as follows: (1) scanning speed was the most relevant SLM parameter regarding the influence on the Ni/Ti ratio, whereas laser power had the least impact and (2) the martensitic transformation temperature variations is monotonic by individually changing scanning speed, laser power, or hatch spacing. It leads to a feasible approach to tailoring the phase transformation temperatures. Similar studies may be found in the work of Haberland et al. [119], where the authors determined an optimum set of parameters for the fabrication of dense Ni-rich NiTi parts with minimum effects of processing on impurity pick-up and on transformation temperatures. Also, Saedi et al. [159] performed a complete study on the effects of laser power and scanning speed on the superelastic response and microstructural properties of a Ni-rich NiTi alloy, concluding that (1) the combination of high (low) laser powder and high (low) scanning speeds leads to fully dense parts since energy input is of key importance for the SLM process, (2) as the energy level increases the texture in [002] direction also increases, and (3) samples fabricated using the same energy level do not behave equally, meaning that all the process parameters have to be investigated meticulously when an optimum process window is aimed. Regarding the last statement of Saedi et al. [159], Yu et al. [160] shared similar conclusions in their study, plus the essential influence of scanning speed on the transformation temperatures—according to the findings of Wang et al. [157]. As in the work of Haberland et al. [119], it was also determined a set of optimum process parameters, in this case regarding a good performance under tensile test (735 MPa) and elongation (10.88%). Some studies have focused on a way to print parts with excellent functional properties in the as-printed condition, eliminating the need for posttreatment (e.g., solubilization or ageing) thus reducing costs and leading time. Xue et al. [161] determined through a computational analytical model a way for predicting the melt pool dimensions in laser powder bed fusion technique, aiming to determine a set of optimum process parameters that delivered fully dense parts of near-equiatomic and Ni-rich NiTi alloys. This approach made it possible to fabricate parts in a wide range of processing parameters using several combinations of parameters. Moreover, excellent mechanical properties were achieved by as-printed specimens, with tensile ductility up to 16%, 6% of strain recovery in shape recovery tests, and 4% in superelastic assessment for the Ni-rich ones. Moghaddam et al. [133]

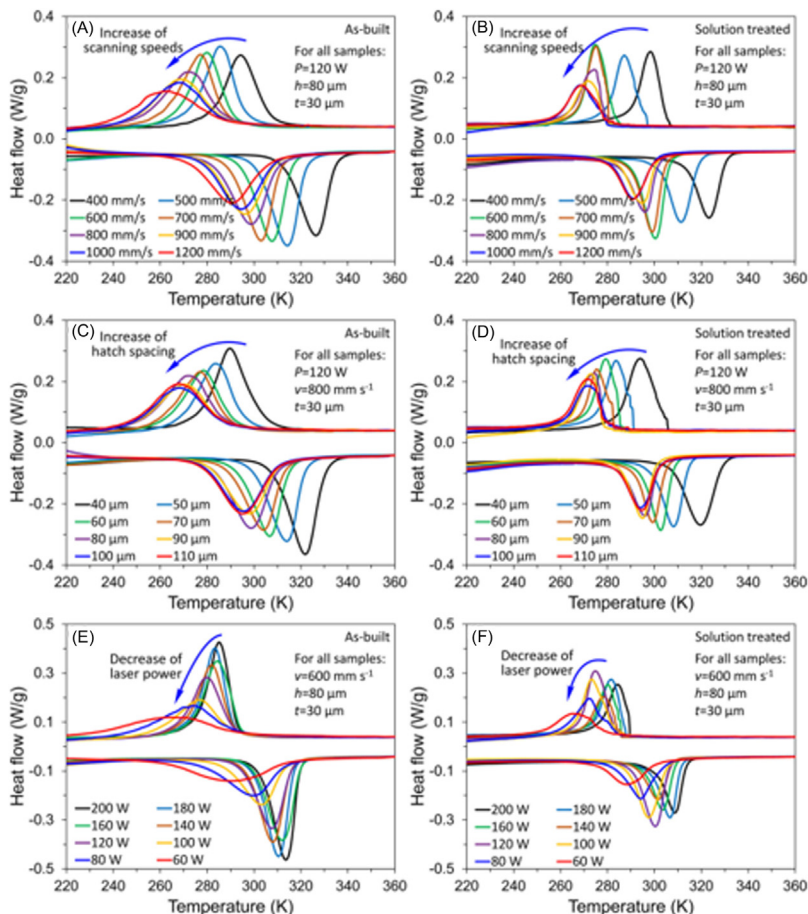


Figure 1.15 Phase transformation behavior of NiTi alloys fabricated by selective laser melting under: (A, B) different scanning speed from 400 to 1200 mm/s ($P = 120$ W and $h = 80$ μ m are fixed); (D, E) different hatch spacing from 40 to 110 μ m ($P = 120$ W and $v = 800$ mm/s are fixed); (G, H) different laser power from 60 to 200 W ($v = 600$ mm/s and $h = 80$ μ m are fixed). The samples presented in (A), (C) and (E) are the as-built samples. The samples presented in (B), (D) and (F) are the as-built samples after solution treatment at 1273 K for 2 h.

Source: From X. Wang, et al., Effect of process parameters on the phase transformation behavior and tensile properties of NiTi shape memory alloys fabricated by selective laser melting, *Addit. Manuf.* 36 (2020) 101545.

focused on the control of processing parameters to enhance the superelasticity of SLMed NiTi through the tuning of microstructure and texture. For this purpose, laser powder (250 W) and scanning speed (1250 mm/s) were kept constant while the hatch spacing (and therefore the energy density—J/mm³) varied from 80 to 180 μ m in regular intervals of 20 mm. It was found that the shortest hatch distance promoted the

finest melt pools due to the intense overlapping between neighboring tracks, leading to higher transformation temperatures and hardness besides grain refinement. The impact of it was reflected on the superelastic behavior and texture illustrated Fig. 1.16A: a recovery rate of 98% and a strain recovery of 5.62% in the first cycle, which when stabilized after 10 cycles resulted in 5.2%. The authors attributed this excellent result to a strong [001] texture (Fig. 1.16B and C) along the build direction, without influence of Ni_4Ti_3 —well known for improving the superelasticity of Ni-rich NiTi alloys [107]—since it was absent.

DED processes, more specifically LENS, have found room for improvement and achieved substantial progress [141,143,162–165]. Baran et al. [141] kept both powder feed rate (5 g/min) and laser power (400 W) for investigating the influence of scanning velocity (1–30 mm/s) on the alloy microstructure and phase composition of a Ni-rich NiTi alloy. Fig. 1.17 exhibits the resulting microstructure, where a gradual transition from axial to columnar grains is seen as the

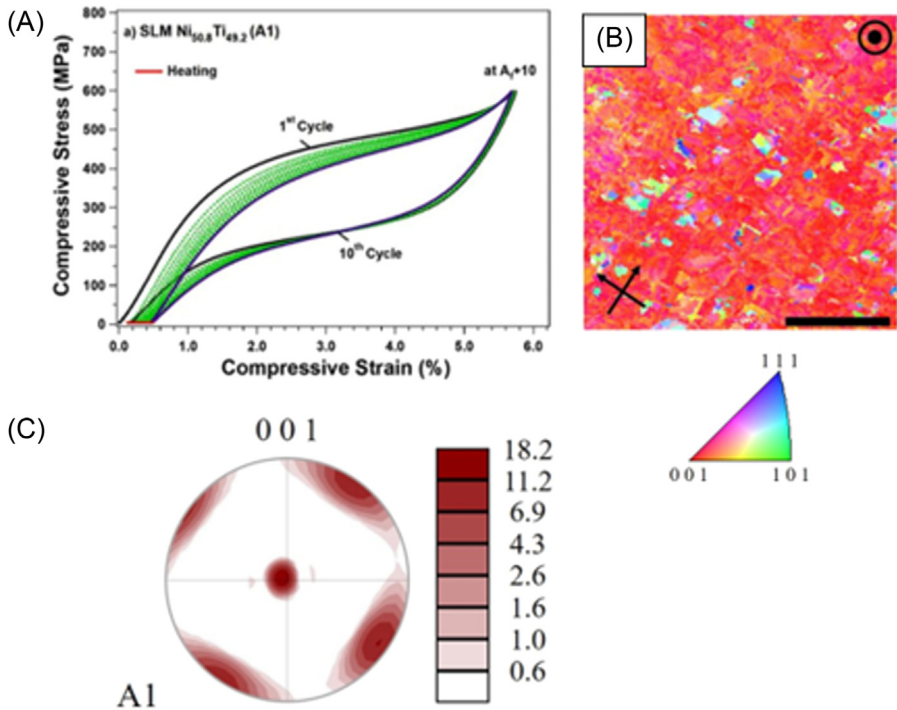


Figure 1.16 (A) Superelastic cycling of SLM Ni50.8Ti 8 (at.%) fabricated with a laser power of 250 W, scanning speed of 1250 mm/s, and hatch spacing of 80 μm, (B) electron backscattering diffraction of the sample cycled in (A) and respective pole figure texture plot. The scale bar in (B) is 300 μm.

Source: From N.S. Moghaddam, et al., Achieving superelasticity in additively manufactured NiTi in compression without post-process heat treatment, Sci. Rep. Revis. 9 (2018) 41.

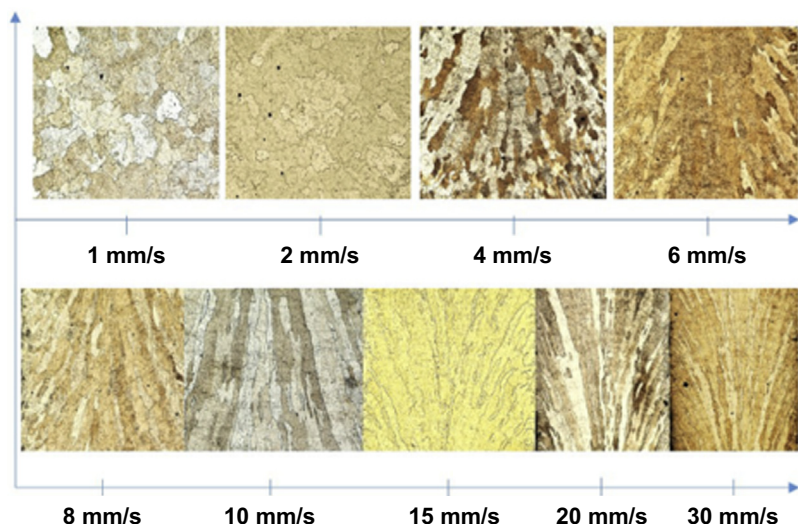


Figure 1.17 Microstructure of the laser deposited samples by LENS using different deposition velocities.

Source: From A. Baran, M. Polanski, Microstructure and properties of LENS (laser engineered net shaping) manufactured Ni-Ti shape memory alloy, *J. Alloy. Compd.* 750 (2018) 863–870.

scanning velocity increased, resulting from an increased energy input level. The energy level impairs directly on the melt pool size, hence on the solidification mode; also, a weaker bonding between the layers results in the reason of the larger melt pool for low energies. The melt pool characteristics were investigated by Gao et al. [162], where a three-dimensional numerical model considering heat transfer (thus solidification rate and temperature gradient), phase change, and fluid flow (Marangoni effect) to simulate the cladding geometry, melt pool features, and deposition rate. An effective comparison between theoretical and experimental melt pool geometries for different parameters of laser power, scanning speed, and deposition rate were possible. Regarding the conclusions of this study, it was summarized that (1) clad geometry and clad height are primarily influenced by laser power and power density per unity area, respectively, (2) scan speed has a little effect on the deposition rate, (3) fluid velocity influences strongly influences the distribution of the elements on the molten pool, with a remarkable influence of the process parameters on the fluid velocity, and (4) temperature gradient and solidification rate influenced directly the grain morphology (as seen by Baran et al. [141] for different scanning velocities).

High-temperature shape memory alloys (HTSMA)—NiTiX ($X = \text{Pt}, \text{Pd}, \text{Au}, \text{Hf}, \text{Zr}$)—are SMA with transformation temperatures above the limited 100°C of the conventional NiTi alloys, reaching 400°C – 700°C depending on its composition. These are of potential application for aerospace, automotive, and energy exploration

industries in reason of their higher operational temperature. However, its poor workability, brittleness, and high materials costs impair their applications. AM has given its first steps showing the feasibility of producing HTSMA [128,166]. NiTiHf alloys have been fabricated by SLM, as demonstrated by the work of Elahinia et al. [128]. In this study, a slightly Ti-rich NiTi-20Hf was printed and compared to the as-extruded alloy. Transformation temperatures of the as-printed samples were slightly lower than the as-extruded, despite above 200°C, in reason of the formation of secondary Ti-rich phases during both atomization and printing processes (i.e., microstructurally). Concerning the two-way shape memory effect, under stress-free thermal cycling response, the HTSMA behavior was similar to the extruded alloy, exhibiting a small magnitude of such effect. The constant-force thermal cycling (compression at 500 MPa) resulted in higher transformation strains for the HTSMA: 1.52% versus 1.26% for the as-extruded. According to the authors, It indicates that a postheat treatment of the SLMed samples may improve this response, as seen in SM/HTSMA [167–169]. Therefore, AM of HTSMA offers outstanding opportunities for further development.

Wang et al. [170] compared in their study the in situ alloying of NiTi (i.e., use of premixed Ni and Ti elemental powders instead of prealloyed NiTi alloy as a feedstock) by three powder-based methods, namely SLM, DED, and EBM. They stated that LENS fabricated the most appropriate parts, followed by SLM and EBM. The negative aspects were based on excessive Ni evaporation and unwanted phases (DED), porosity and inhomogeneous microstructure (SLM), and powder combustion (EBM). Nonetheless, it was stated that high-quality solid parts are achieved using DED by carefully adjusting the Ni:Ti ratio. The biomedical behavior has been extensively reported on AM of NiTi-based SMAs due to their potential for a wide array of applications in this field. According to Dadbakhsh et al. [118], one reason for that is the ability to manipulate the stable phase to engineer components (e.g., scaffolds). The same is valid for the required solid volume fraction or porosity, which are adjusted considering the load-bearing applications of the final implant. Also, since cell proliferation is favorable in regions of smooth curvatures, porous design plays a central role; then powder characteristics and laser parameters are equally important. Therefore, the degrees of freedom provided by AM allows biomedical devices to be designed with maximum biomechanical and physiological compatibility for a specific patient, reducing either the chance of implant rejection or/and the patient healing period, improving the lifetime performance of the implant. As an illustration, Fig. 1.4 depicts SLM NiTi specimens with different porosities and different ways of production by varying process parameters and print configurations, that is, different strategies. The parts in Fig. 1.4A and B have the same porosity (99.1%) but were printed in parallel (A) and perpendicularly (B) to the printing bed. In the case of Fig. 1.4C, the density decreased to 88.5% due to the larger hatch distance if compared to the previous examples; in Fig. 1.4D the model was designed aiming at open porosity, leading to a density of 65.4%. Fig. 1.4E–H depicts the correspondent fluorescence micrographs of the aforementioned specimens; after 8 days of cell culture, all surfaces were covered with a layer of viable cells. This fact demonstrates the

biocompatibility of the SLMed implant material and the attractiveness of this method for producing NiTi implants.

1.4.3 Processing of NiTi-based alloys: fields of application

The biomedical behavior has been extensively reported on AM of NiTi-based SMAs due to their potential for a wide array of applications in this field. According to Dadbakhsh et al. [118], one reason for that is the ability to manipulate the stable phase to engineer components (e.g., scaffolds). The same is valid for the required solid volume fraction or porosity, which are adjusted considering the load-bearing applications of the final implant. Also, since cell proliferation is favorable in regions of smooth curvatures, porous design plays a central role; then powder characteristics and laser parameters are equally important. Therefore, the degrees of freedom provided by AM allows biomedical devices to be designed with maximum biomechanical and physiological compatibility for a specific patient, reducing either the chance of implant rejection or/and the patient healing period, improving the lifetime performance of the implant. As an illustration, Fig. 1.18 depicts SLM NiTi specimens with different porosities and different ways of production by varying process parameters and print configurations, that is, different strategies. The parts in Fig. 1.18A and B have the same porosity (99.1%) but were printed in parallel (A) and perpendicularly (B) to the printing bed. In the case of Fig. 1.18C, the density decreased to 88.5% due to the larger hatch distance if compared to the previous examples; in Fig. 1.18D, the model was designed aiming at open porosity, leading to a density of 65.4%. Fig. 1.4E–H depicts the correspondent fluorescence micrographs of the aforementioned specimens; after 8 days of

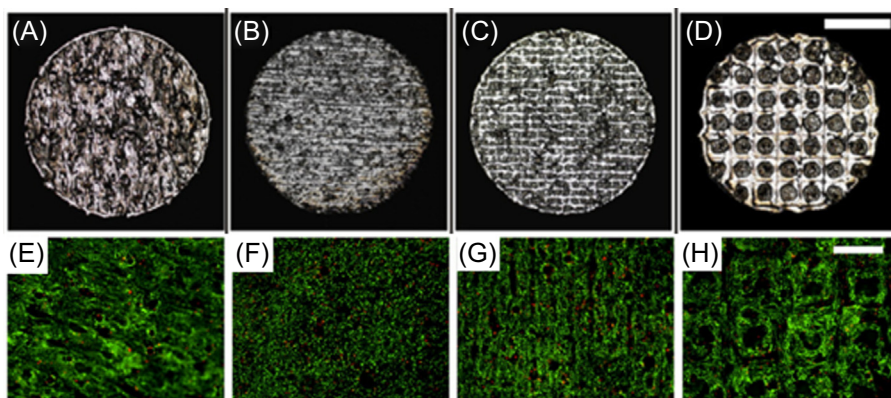


Figure 1.18 (A–D) NiTi specimens fabricated by SLM using different printing strategies and models, (E–H) correspondent fluorescence micrographs showing living cells (green) with a negligible minority of dead cells (red) in fluorescence micrographs after culturing human mesenchymal stem cells for eight days. The bottom fluorescence images are related to the upper specimens. The scale bar of (A–D) is 3 mm, whereas (E–H) is 1 mm. *Source:* From T. Habijan, et al., The biocompatibility of dense and porous nickel-titanium produced by selective laser melting, Mater. Sci. Eng. C 33 (2013) 419–426.

cell culture, all surfaces were covered with a layer of viable cells. This fact demonstrates the biocompatibility of the SLMed implant material and the attractiveness of this method for producing NiTi implants.

Other than biomedical applications, microelectromechanical components are also suitable for being fabricated by AM. Following the trend, Clare et al. [171] fabricated cantilever beams of NiTi by SLM. It was shown that the laser power affected the resultant phases in the built component. Hence, as the power increased, a mixture of martensite and rhombohedral R-phase were identified utilizing X-ray diffraction. Interestingly, R-phase is responsible for a gradual phase transition (austenite $\rightarrow$ R-phase $\rightarrow$ martensite), which in turn is beneficial for this components' application. Elahinia et al. [172] also manufactured by SLM a porous SMA actuator. In this case, this Ti-rich actuator did not require shape settings, since the memorized shape is the printed one. Moreover, besides the simple fabrication method, due to the porous structure, the lightweightness was assured. Dudziak et al. [173] demonstrated the feasibility of producing microactuators by SLS. In this work, fine NiTi powder was processed using SLS aiming to generate structures in the micrometer range. As a result, the shape memory effect was preserved within a reasonable field of processing parameters, and it was possible to correlate the shift of transition temperature with the process parameters.

Lastly, functionally graded structures have been widely explored in reason of the possibility for tailoring transformation temperatures during the printing processes; then, parts composed of both martensite and austenite are sequentially layered in as-printed condition. Both LENS [163,174] and SLM [134,175,176] were employed to explore such possibility. For illustrating, Wang et al. [134] reported a structure built by SLM with remarkable damping properties at low (1 Hz) and high (90 Hz) frequencies, plus the Elinvar effect (or the compensation of variation of Young's modulus during the thermal cycle) in reason of the gradual transformation of martensite to austenite.

1.4.4 Processing of other SMA alloys by powder-based additive manufacturing

Concerning the processed alloys, mostly are NiTi-based alloys in reason of their superior mechanical response, biocompatibility, and lightweightness. However, AM has been explored for successfully printing the following SMAs systems: Cu-Al-Ni-(Mn) [115,124,131,137], Fe-Mn-Al-Ni [129], and Ni-Mn-Ga [143,148,149]. Copper-based alloys are suitable for sensors and actuators at temperatures around 200°C due to their high thermal stability, although suffering from brittleness and exhibit low percentage strain recovery. Gustmann et al. [124] studied the influence of process parameters on the SLM of Cu-Al-Ni-Mn alloy, reaching up to 99% of density and fully martensitic samples with energy input between 30 and 40 J/mm³. Iron-based alloys are cheaper if compared to both copper and nickel-based alloys, besides being easier to fabricate (based on conventional steel making processes). Besides a lesser shape memory capacity and a large transformation hysteresis seems

to limit their application [177,178], AM has shown its capability to circumvent these problems. Niendorf et al. [129] and Ferretto et al. [179] printed successfully by laser powder bed fusion Fe-Mn-Al-Ni and Fe-Mn-Si, respectively. The former authors achieved good pseudo-elasticity (a maximum of 7.5% of reversible strain at an applied compressive strain of about 11%) after single-step heat treatment. Ferretto et al. [179] stated that a high energy density (194.44 J/mm^3) was necessary to ensure satisfactory samples without defects. These presented high strength and ductility (47.86% of fracture elongation) after heat treatment, with superior performance if compared to the specimens conventionally fabricated and containing VC precipitates. Lastly, nickel-based can exhibit both magnetocaloric as well as magnetic shape memory effects, hence being known also as magnetic shape memory alloys (MSMA). Despite its giant magnetic-field-induced strain (MFIS), bulk alloys are brittle, the fabrication process is expensive and MFIS is moderate in reason of high twinning stresses, limiting their practical. High-entropy alloys (HEAs) are novel in the metal materials community [180–184]. The selection of the proper combinations of their constituent elements and their proportions leads to a high mixing configurational entropies that promote the stabilization of solid solutions based on face-centered cubic (fcc), body-centered cubic (bcc) or hexagonal close-packed (hcp) structures [185–187] application [150]. AM arises as an alternative, and Ni-Mn-Ga alloy was printed by both binder jetting [188,189], and 3D-printed inks containing the elemental powders [149]. Fig. 1.19 illustrates the MFIS of porous Ni-Mn-Ga obtained by Caputo et al. [188] during the first and third heating/cooling cycles. It shows a reversible MFIS strain of $\sim 0.01\%$, indicating the feasibility of AM for printing magnetic shape memory alloys.

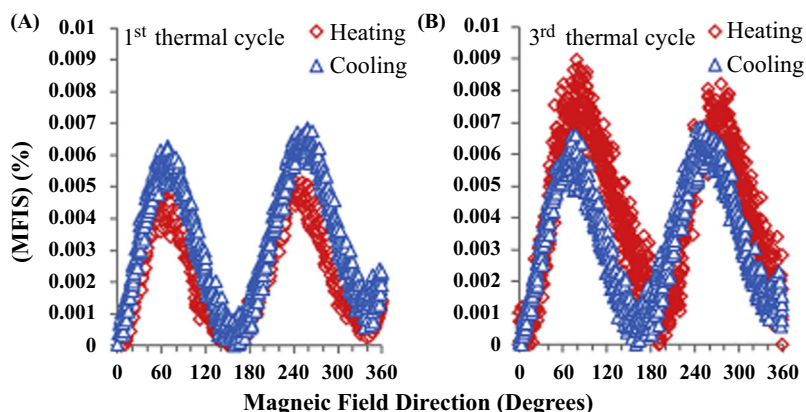


Figure 1.19 MFIS as a function of magnetic field direction for a sintered sample obtained by binder jetting of Ni-Mn-Ga powders recorded during the (A) first and (B) third heating/cooling cycles.

Source: From M.P. Caputo, A.E. Berkowitz, A. Armstrong, P. Müllner, C.V. Solomon, 4D printing of net shape parts made from Ni-Mn-Ga magnetic shape-memory alloys, Addit. Manuf. 21 (2018) 579–588.

1.5 Powder-based additive manufacturing of high-entropy alloys

1.5.1 Introduction

High-entropy alloys (HEAs) are novel in the metal materials community [180–184]. The selection of the proper combinations of their constituent elements and their proportions leads to a high mixing configurational entropies that promote the stabilization of solid solutions based on face-centered cubic (fcc), body-centered cubic (bcc) or hexagonal close-packed (hcp) structures [185–187]. Fig. 1.20 illustrates the strengthening

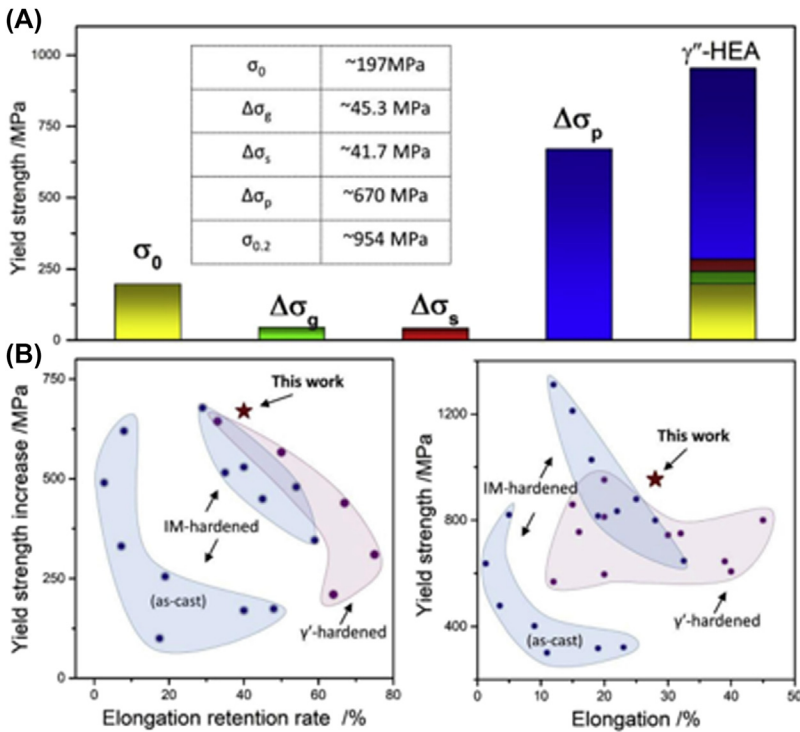


Figure 1.20 Example of a HEA with superior strength due to both ordering strengthening and coherency strengthening from the work of He et al. [185] for a $\text{Ni}_2\text{CoCrFeNb}_{0.15}$. (A) Contributions of different strengthening mechanisms; (B) the comparative results of yield strength increase and ductility retention; (C) ashby plot showing the advantage of γ'' strengthened HEAs. Mechanical properties obtained from CoCrFeNiMnAl [190], $\text{CoCrFeNiAl}_{0.5}$ [191], $(\text{CoCrFeNi})_{94}\text{Ti}_2\text{Al}_4$ [192], $(\text{CoCrNi})_{94}\text{Ti}_3\text{Al}_3$ [193], $\text{Al}_{0.2}\text{Co}_{1.5}\text{CrFeNi}_{1.5}\text{Ti}_{0.3}$ [194], $\text{Al}_{3.31}\text{Co}_{27}\text{Cr}_{18}\text{Fe}_{18}\text{Ni}_{27.27}\text{Ti}_{5.78}$ [195], $(\text{FeNi})_{67}\text{Cr}_{15}\text{Mn}_{10}\text{Al}_{8-x}\text{Ti}_x$ [196], $\text{Al}_{10}\text{Co}_{25}\text{Cr}_8\text{Fe}_{15}\text{Ni}_{36}\text{Ti}_6$ [197], $\text{CoCrFeNiMo}_{0.3}$ [198], and $\text{Cr}_{15}\text{Fe}_{20}\text{Co}_{35}\text{Ni}_{20}\text{Mo}_{10}$ [199].

Source: From F. He, et al. Design of D022 superlattice with superior strengthening effect in high entropy alloys, *Acta Mater.* 167 (2019) 275–286.

contributions of a novel $\text{Ni}_2\text{CoCrFeNb}_{0.15}$ [185]. HEAs can exhibit exceptional strength, ductility and fracture toughness at cryogenic temperatures, as well as superconductivity, superparamagnetism, and exceptional irradiation resistance [184,200–205]. Fig. 1.21 [202] illustrates the notable high strength and ductility for low-SFE HEAs compared with other conventional alloys. Recent substantial reviews have reported on HEAs [200,202,209–214]. Excellent reviews of the recent advances on HEAs for 3D printing was written by Han et al. [215] and Moghaddam et al. [216].

1.5.2 Technological overview

The novel properties of HEAs, such as high mechanical performance at high temperature, excellent specific strength, exceptional ductility and fracture toughness at cryogenic temperatures, superparamagnetism and superconductivity, can be explored in various applications in transportation, energy, aerospace, biomedical electronics, and tools sectors. Hydrogen storage materials, radiation-resistant materials, thermal-sprayed, hard low-friction and biomedical coatings, precision resistors, diffusion barriers for electronics, electromagnetic shielding materials, binders, and soft magnetic and thermoelectric materials are the possible applications of HEAs [217–222]. Direct energy deposition (DED) and powder bed fusion (PBF) is the primary 3D printing techniques used for HEAs [215]. The ultrafast cooling rates in 3D printing prevents the formation of undesired intermetallic compounds.

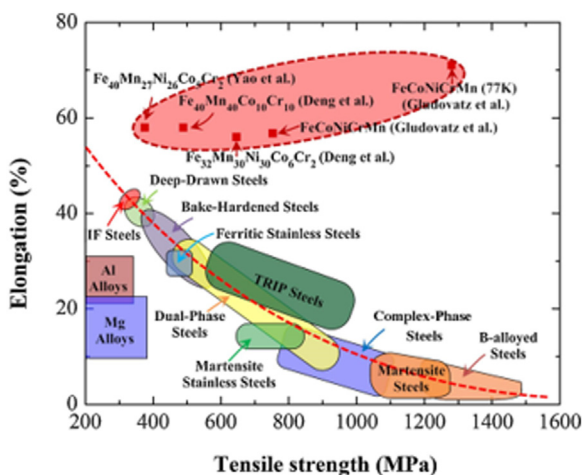


Figure 1.21 Example of the notable high strength and ductility for low-SFE HEAs: $\text{Fe}_{40}\text{Mn}_{27}\text{Ni}_{26}\text{Co}_3\text{Cr}_2$ [206], $\text{Fe}_{40}\text{Mn}_{40}\text{Co}_{10}\text{Cr}_{10}$ [207], $\text{Fe}_{32}\text{Mn}_{30}\text{Ni}_{30}\text{Co}_6\text{Cr}_2$ [207], and FeCoNiCrMn [204] at room temperature and FeCoNiCrMn [204] at 77K, compared with other conventional alloys [208].

Source: From Y.F. Ye, Q. Wang, J. Lu, C.T. Liu, Y. Yang, High-entropy alloy: challenges and prospects, *Mater. Today* 19 (2016) 349–362.

1.5.3 Powder for HEA development

Prealloyed powders developed by gas (atomization Fig. 1.22 [223]), water atomization, or mechanical alloying can be used in the 3D printing of HEA. In addition, elemental powders through in situ alloying can also be used in DED and PBF techniques. The constituent elements and their proportions determine the phases of gas-atomized HEA powders [224,225].

Mechanical alloying is a high-energy ball milling process to develop fine metal powders for 3D printing [226–228] and is a promising technique to produce homogeneous microstructures of HEA powder particles with increased solid solubility [229,230] Fig. 1.23. An example of particles of CoCrFeMnNi high-entropy alloys produced by mechanical alloying is shown in Fig. 1.23 [231]. Higher degrees of sphericity, lower oxygen content, and better printability compared with those produced by water atomization and mechanical alloying are the characteristics of HEA powders produced by gas atomization, and it is the most popular method to develop HEA powders for PBF and DED printing processes, despite the high cost and low production efficiency.

Mechanical alloying can conveniently realize the alloying of any ratios of elemental powders in contrast to gas atomization and water atomization. However, mechanical alloying compromises the sphericity of powders, resulting in poor flowability. Spheroidization techniques [232,233] using plasma and pulsed electron beam irradiation (Fig. 1.24 [233]) can convert powders with an initial irregular shape to a spherical shape, improving the flowability and printability of mechanically alloyed HEA powders for 3D printing.

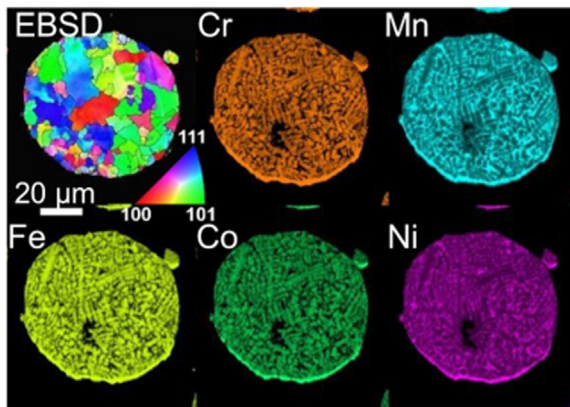


Figure 1.22 EBSD image and the corresponding elemental distribution of the prealloyed equiatomic CoCrFeNiMn HEA powder produced using vacuum induction melting gas-atomization with argon gas. A dendritic structure enriched in Fe, Cr, and Co is present. *Source:* From P. Wang, et al. Additively manufactured CoCrFeNiMn high-entropy alloy via pre-alloyed powder, *Mater. Des.* 168 (2019) 107576.

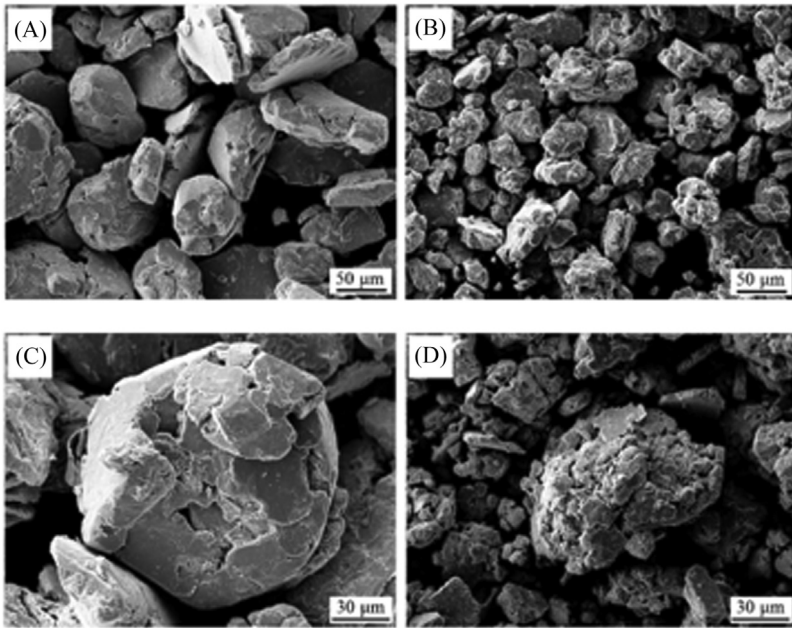


Figure 1.23 SEM images of particles of CoCrFeMnNi high-entropy alloys produced by mechanical alloying after high-energy ball milling in a planetary ball mill using 5-mm diameter zirconia balls in argon gas for (A, C) after 20 min and (B, D) 60 min.

Source: From S.-H. Joo, et al., Structure and properties of ultrafine-grained CoCrFeMnNi high-entropy alloys produced by mechanical alloying and spark plasma sintering, *J. Alloy. Compd.* 698 (2017) 591–604.

1.5.4 Techniques for 3D printing of HEAs

DED exhibits the great capability to develop functionally graded HEAs through in situ alloying using elemental powders. [Table 1.4 \[216\]](#) summarizes the DED processing parameters and the characteristics of fabricated HEAs. Borkar et al. [\[287\]](#) investigated DED-printed graded $\text{Al}_x\text{CrCuFeNi}_2$ ($0 \leq x \leq 1.5$) and $\text{AlCo}_x\text{Cr}_{1-x}\text{FeNi}$ ($0 \leq x \leq 1$) HEAs [\[290\]](#). The printed $\text{Al}_x\text{CrCuFeNi}_2$ HEAs achieved a transition in microstructure from a disordered FCC to FCC + ordered L_{12} to disordered BCC + ordered B2 with the increase in Al content. Döbelstein et al. [\[291\]](#) developed by DED crack-free graded refractory $\text{Ti}_{25}\text{Zr}_{50-x}\text{Nb}_x\text{Ta}_{25}$ HEAs with x from 0 to 50. Moreover, graded products from bulk metallic glass to HEAs were developed by Welk et al. [\[282\]](#). Refractory HEAs using DED were also developed, such as MoNbTaW [\[283\]](#), TiZrNbHfTa [\[292\]](#), and TiZrNbMoV [\[293\]](#). Novel HEA composites have been developed using DED, such as AlCoCrFeCu with the addition of Y_2O_3 partially stabilized ZrO_2 , preventing microcracks and promoting the refinement of the microstructures [\[294\]](#). Moreover, CoCrFeMnNi with TiC [\[295\]](#) and WC [\[296\]](#) was developed to improve its tensile strengths.

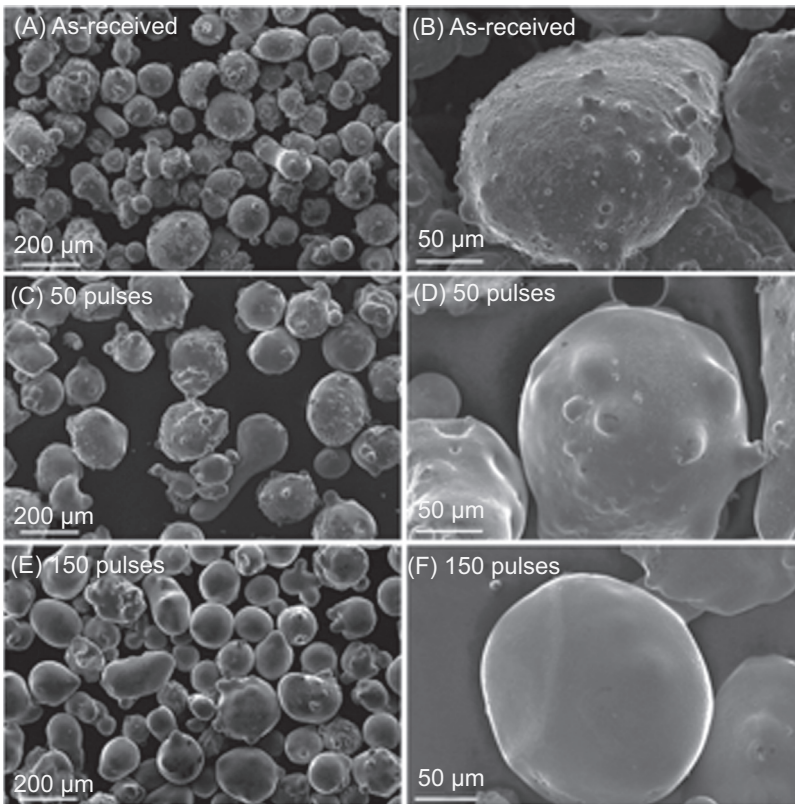


Figure 1.24 Example of the spheroidization of CoCrW using pulsed electron beam technique. The powder surface asperities up to 20 μm size can be eliminated by melting and incorporation into the near-surface of the particle. The powder was subjected to 50 pulses and 150 pulse irradiation at 40 kV.

Source: From J.W. Murray, M. Simonelli, A. Speidel, D.M. Grant, A.T. Clare, Spheroidisation of metal powder by pulsed electron beam irradiation, *Powder Technol.* 350 (2019) 100–106.

PBF has been used to print HEA due to its capability to produce complex components with outstanding mechanical properties [10,280]. Table 1.5 [216] summarizes the PBF processing parameters and the characteristics of fabricated HEAs. PBF-printed CoCrFeNi HEA was first developed by Brif et al. [323]. Multiple grains that grew epitaxially from existing grains in the substrate, with cell axes perpendicular to the fusion line, are characteristic of a single track of CoCrFeMnNi [324]. Johnson et al. [301] proposed a melt pool geometry-based criteria for predicting the printability of a CoCrFeMnNi HEA produced by PBF. The effect of the laser energy density on PBF-printed CoCrFeMnNi and AlCoCrFeNi HEA [264,306] showed that the increase in energy density eliminated porosity and avoided delamination between layers. High cooling rates in PBF promoted the

Table 1.4 Summary of DED processing parameters and the characteristics of fabricated HEAs.

HEAs	Laser power (W)	Scanning speed (mm/min)	Beam size (Hatch spacing) (mm)	Feed rate (g/min)	Characteristics	References
HEAs claddings						
CoCrCuFeNi, preplaced	2000	400	4.5	—	fcc, equiaxed to dendritic transition, nanoprecipitates inside the dendrites, disordered-ordered transition in interdendrites	[234]
CoCrCuFeNi					fcc, dendritic (Cr rich) + interdendritic (Cu rich) structure	[235]
CoCrFeMnNi	1400	240	4	8	fcc, no pores or cracks, ellipsoidal dendrite and interdendrite structures, high temperature softening resistance	[236]
CoCrFeNiB _x preplaced	1700	360	2.4	—	fcc + borides	[237]
CoCr _x FeNiB preplaced	1200	180	2.5	—	fcc + borides	[238]
Al ₃ CoCrFeNi	2000	300	4.5		bcc + minor fcc fine equiaxed dendritic	[239]
FeNiCoSiCrAlTi	2000	400	4.5		bcc, equiaxed polygonal grains, interdendritic regions	[240]
Al _x CoCrCuFeNi (1 ≤ x ≤ 2)	1200 – 2000	120 – 720			bcc + fcc, the hardness and abrasion resistance increased with increasing Al content	[241]
AlCoCrFeNi	1000, 1200	6000	0.6		bcc ₁ + bcc ₂ + Al ₃ Ni + FeAl ₃ ,	[242]

(Continued)

Table 1.4 (Continued)

HEAs	Laser power (W)	Scanning speed (mm/min)	Beam size (Hatch spacing) (mm)	Feed rate (g/min)	Characteristics	References
AlCoCrFeNi	600 – 650	300	2.3	5.6	fcc + bcc, 3-layer cladding, Ni-depletion	[243]
Al _x CoCrFeNi	800 – 1200	400 – 1200	3(0.75 – 1)		fcc ($x = 0.3$), fcc + bcc ($x = 0.6$), bcc ($x = 0.85$) grain boundary segregation and substructural cellular networks	[244]
Al _{0.8} CoCrCu _x FeNi ($0 \leq x \leq 1$)	1850	120	1.2		bcc ₁ + B2 ($x = 0$), bcc ₁ + B2 + fcc ($x \geq 0.25$), Cu reduces the cracking sensitivity	[245]
AlCoCrNiTiV, preplaced on substrate	2000	2400	3		B2 matrix and (Co, Ni)Ti ₂ compounds with few Ti phases	[246]
AlCoCuFeNi	800 – 2000	600	3	3.3	fcc + bcc, Fe-rich bcc interdendritic region and Cu-rich fcc dendritic region	[247]
AlCoCrFeNiTi	2000	300	4.6		bcc + B2 + Ti-rich intermetallic, dendritic microstructure	[248]
AlCoCrCu _x FeNiSi _{0.5} Preplaced on substrate	2000	600	4		bcc + fcc, Cu-poor dendritic bcc, Cu-rich interdendritic fcc	[249]
Al ₂ CrFeNiMox ($0 \leq x \leq 2$)	900	240	4		bcc ₁ + bcc ₂ , equiaxed grains, eutectic structure in the interdendritic regions at Mo	[250]
Al _{1.8} CoCrCu _{0.7} FeNiB _{0.3} Si _{0.1} preplaced	2000	400	4.5		bcc, thermally stable up to 1000°C	[251]

AlCoCrCu_{0.5}FeNiSi preplaced	3000	600	4		bcc, fine dendritic structure with nanoprecipitates	[252]
TiZrNbWMo sprayed	3000	400	2.3		bcc, dendritic (rich in W) and interdendritic (rich in Nb, Mo β -Ti _x W _{1-x} precipitates structures	[253]
CrMoTaWZr sprayed	400 – 1100	6000	0.6		bcc ₁ (rich in Cr, Ta, Zr) + bcc ₂ (rich in Mo, Ta, W) + Laves phase ((Cr/Ta) ₂ Zr)	[254]
MoFe_{1.5}CrTiWAlNb_x (x = 1.5, 2, 2.5, 3)	3000	240			bcc + (Nb, Ti)C carbides + C14-Laves, cellular (x ≤ 2) and columnar (x > 2) microstructures	[255]
AlTiVMoNb preplaced	3700	600			bcc, dendritic (rich in Ti), interdendritic (rich in Al, Nb, Mo)	[256]
AlCrSiTiV	2000	180	2.5 (1.5)	2	bcc + (Ti,V) ₅ Si ₃	[257]
CoFeNi₂Nb_xV_{0.5} (x = 0.75, 1)	1400 – 1800	240	4		fcc + Fe ₂ Nb-type Laves, cellular dendritic	[258]
B_{3.25}Co_{14.5}Cr₃₂Fe₃₆Ni₁₀Si_{4.25}	467	100	2.3		fcc- γ (Fe, Ni) + bcc-CoFe _{15.7} + amorphous phase (~ 49%)	[259]
B₁₄Co₃₄Cr₂₉Fe₈Ni₈Si₇	520	100	2.2		Amorphous layer + β -Co phase, layered microstructure	[260]
AlCoCrCu_{0.9}FeNi	3000	600	4		bcc, Cu-rich precipitates, high corrosion resistance	[261]
AlCoCrCuFeNi	300	120	1		bcc + fcc, a beneath Cu-diluted layer	[262]

(Continued)

Table 1.4 (Continued)

HEAs	Laser power (W)	Scanning speed (mm/min)	Beam size (Hatch spacing) (mm)	Feed rate (g/min)	Characteristics	References
Bulk HEAs CoCrFeMnNi	1700 1000 – 1400	120 400, 500		10 7 – 9	fcc, No segregation fcc, anisotropic properties at 1000, 1200 W, isotropic properties at 1400 W, No segregation	[263] [264,265]
	880	600	2.5 (1.2)	8.6	fcc, Mn and Ni segregation in interdendritic region	[266]
	600 – 1000 400	800 300	2 (1) 0.6 (0.46)	10	fcc, low porosity, cellular morphology, Mn and Ni enriched intercellular	[267] [268]
	350–400 1000 – 1400	400 – 600 400				
CoCrFeNiMo_{0.2}				7 – 9	fcc, columnar grains morphology, larger columnar grains at 1400 W, better corrosion resistance than 304/316 L	[269] [270]
CoCrFeNiNb_x	1600 – 1650	480	3 (1.5)		fcc + laves phase (hcp), excellent printability, (CrFe) (CoNi)Nb type Laves phase was rich in Nb and poor in Cr and Fe	[77]
CrCoNi	380 – 400	846	– 0.4	2.52	fcc, few pore defects, columnar grain	[271]
AlCoCrFeNi		150 – 2400	0.15		bcc-type (disordered A2 + ordered B2)	[272]

AlCoCrFeNi	80 KJ	1200			bcc, dendrite (50 – 300 μm) and interdendrite phases,	[273]
Al_{0.3}CoCrFeNi	300	1020	0.5 (0.381)		fcc, formation of Al–Ni rich solute nanoclusters during DLD, and nano L1 ₂	[274]
AlCoCrFeNi_{2.1}	900	875		30	Ordered L1 ₂ + disordered bcc, L1 ₂ dendrites and L1 ₂ + bcc eutectics phases,	[275]
Al_xCoCrFeNi ($x = 0.3, 0.6, 0.85$)	800	800	4		fcc ($x = 0.3$), fcc + bcc ($x = 0.6$), bcc ($x = 0.85$), Al segregation in grain boundaries ($x = 0.3$), Al and Ni enriched interplates ($x = 0.6$), Fe and Cr segregated into cuboidal particle phase, and Ni and Al segregated into matrix phase ($x = 0.85$)	[276]
Al_xCoCrFeNi_{2-x} ($x = 0.3, 1$)		600	0.25		Al _{1.7} FeCoCrNi _{0.3} (A2 + B2), AlFeCoCrNi (A2 + B2 + L1 ₂)	[277]
Al_{0.3}CoCrFeN					fcc, large columnar grains, strong texture development, asymmetric tension and compression deformation	[278]
Al_xCoCrFeNi ($x = 0.3, 0.7$)	250 – 300	900		1.5	Oxidation resistance: $x = 0.7 > x = 0.3$, formation of an exterior Cr ₂ O ₃ scale with a beneath Al ₂ O ₃ subscale	[279]
AlCrCuFeNi	1600 – 2200	1200			fcc + bcc, dendritic microstructure	[280]

(Continued)

Table 1.4 (Continued)

HEAs	Laser power (W)	Scanning speed (mm/min)	Beam size (Hatch spacing) (mm)	Feed rate (g/min)	Characteristics	References
Al_xCoCrFeNi ($0.51 \leq x \leq 1.25$)	200	762			fcc + bcc ($x < 1$)), bcc/B2 ($x = 1$) Cellular microstructure, severe lattice distortions associated with the addition of Al	[281]
MoNbTaW	S 1(400), S 2(4500)	150, 500			Cracking due to the brittle nature of MoNbTaW, deviated stoichiometry from the starting elemental composition	[282]
TiZrNbHfTa	2900		3	2.3	bcc, equiaxed grain, crack-free cylinder (diameter: 3 mm, length: 10 mm)	[283]
MoNbTaW_x					bcc, grain size: 20 μm , dendrite size: 4 μm	[284]
NbMoTa					bcc, porosities and intergranular cracks	[285]
MoNbTaW	800 S1 1000 R	254 S1 1778 R	0.6 (0.381)		bcc + minor inclusion of unmelted particles	[286]
Compositionally graded HEAs						
Al_xCoCrFeNi₂ ($0 \leq x \leq 1.5$)	Nd: YAG laser: 500 W, near-infrared laser radiation at a wavelength of 1.064 μm			Variable	fcc ($x \leq 0.4$), fcc + bcc ($x \geq 0.8$), dendritic structure, segregation of Cu into interdendritic regions	[233]

AlCo_xCr_{1-x}FeNi (0 ≤ x ≤ 1)	200–300	400			bcc + B2, spinodal decomposition with decreasing x, a second grain boundary precipitate fcc phase at x = 0.6, 0.8	[287]
Al_xCoCrFeNi (0.3 ≤ x ≤ 0.7)	300	400	0.5		fcc (x = 0.3), fcc + B2 (x = 0.7), anisotropic grains, B2 precipitates at fcc grain boundaries, eutectic lamellar morphology	[288]
AlCrFeMoV_x (0 ≤ x ≤ 1)	400	1020	0.381		bcc, equiaxed to elongated grains with increasing V, hardness increased with increasing V	[289]
Nb_{50-x}Ti₂₅Ta₂₅Zrx (0 ≤ x ≤ 50)	500 – 2500	100 – 600	0.4		bcc, coarse grain (x ≤ 25), bcc + Ta-rich-bcc ₂ , equiaxed grains (x > 25)	[290]

S, Step; R, remelting.

Source: From A. Ostovari Moghaddam, N.A. Shaburova, M.N. Samodurova, A. Abdollahzadeh, E.A. Trofimov, Additive manufacturing of high entropy alloys: a practical review, J. Mater. Sci. Technol. 77 (2021) 131–162.

Table 1.5 Summary of processing parameters, relative density, phase, and defects in PBF-printed HEAs.

HEAs	P (W)	v (mm s ⁻¹)	h (μm)	t (μm)	VED (J/mm ³)	Relative density (%)	Phase	Defect	References
CoCrFeNi	200	300	—	50, 20	—	—	fcc	Porosity	[297]
	60, 90, 120, 150	100, 300, 600, 900, 1200, 1500	50	20	100–1500 (250)	Porosity (%): 1–11.8 (0.09)	fcc + bcc	Porosity	[298]
Co _{1.5} CrFeNi _{1.5} Ti _{0.5} Mo _{0.1}	110–280	800–2000	45–75	30	(40)–(104.3)	(7.5)–(8.12)	—	Micropores	[299]
	160–270 (160)	540–1350 (650)	80–120 (100)	40	–61.5	–99.3	fcc + IMCs	—	[300]
	400	800–4000	90	30	74	98.2	fcc	Cracks and pores	[301]
	160–290 (240)	1500–2500 (2000)	50	40	–60	–99.2	fcc	—	[296]
CoCrFeMnNi	110–280	800–2000	45–75	30	(40)–(259.3)	(7.1)–(7.89)	fcc	Micropores	[299]
	90	600	80	25	75	—	fcc	—	[302]
	400	(800), 1200, 2000, 2500	90	30	(185.2) 123.46, 74.07, 59.26	—	fcc	Irregular pores, grainboundary cracks, Defects increased at higher scanning speed	[303]
	150	500	50	60	100	—	fcc	—	[304]
AlCoCrFeNi	250–(370)	500–2500 (1500)	80	40	62.5–115.6 (77.08)	–99.5	—	Micropores	[305]
	250–400	1000	90	40	69.4, 83.3, (97.2) 111.1	–98.4	A2 + B2	Porosity	[306]
Al _{0.5} CoCrFeNi	98	2000	52	20	47.1	—	A2 + B2	Cracks + porosity	[297]
	400	1600	90	40	69.4	—	fcc	No defects	[307]
	160–(320)	400–2000 (800)	(60)–80	50	–133.3	–99.92	fcc + bcc	No defects at optimized parameters	[308]
Al _{0.3} CoCrFeNi	150–170	1100–1300	45	25–30	85–137	99.9	fcc	No Defects	[309]
Al _{0.26} CoFeMnNi	120, 200	350–650	60–90	30	68.4–246.9	99.5	fcc	—	[310]

AlCoCrCuFeNi	160–400	400–1600	90	40	69.4	7.058	B2 + A2 + fcc	Microcracks + micropores, increased with increasing VED	[311]
AlCoCuFeNi	205	1000	40	30	170.8	—	B2 (printed), B2 + fcc (Anneal)	No cracking or porosity	[312]
			60		208.3	99.82		Spherical pores + vertical MCs	
			80		156.25	99.8		Narrow MCs	
			100		125	99.63		Long MCs	
AlCrCuFeNi	300	400	120	40	104.17	99.6	A2 + B2	Spherical pores + vertical MCs	[313]
					234.4	99.7		Narrow	
			80		156.25	99.8		microcracks	
					117.2	99.5		Irregular pores +	
					93.75	99.3		Microcracks	
					78.13	99		No microcracks for $x = 3$	
AlCrCuFeNi_x ($x = 2.0, 2.5, 2.75, 3.0$)	200	400	80	20	312.5	99.7	fcc + B2, $x = 3$		[314]
AlCrFeNiV	140	900	50	—	—	99.88	fcc + nano L1 ₂	—	[315]
AlCoFeNiSm_{0.1}V_{0.9}	200	10	300	200	333.3	—	fcc	No porosity, No significant crack	[316,317]
AlCoFeNiSm_{0.05}TiV_{0.95}Zr	400	250	100	100	160	—	bcc	—	[318,319]
MoNbTaW	375–325	250–600	100	100	—	—	bcc	No crack	
Ni₆Cr₄WFe₉Ti	300	2500	80	60	25	—	fcc + unknown phase	Cracks + micropores	[320]
	350	2000	70	60	33.3	—			[321]
Fe₄₀Mn₂₀Co₂₀Cr₁₅Si₅	140	800	100	40	43.75	—	ε -hcp + γ -fcc	No cracks, small void (vol. % = 0.1)	[322]

V, Scanning velocity; h, hatch distance; VED, volumetric energy density; IMCs, intermetallic compounds.

Source: From A. Ostovari Moghaddam, N.A. Shaburova, M.N. Samodurova, A. Abdollahzadeh, E.A. Trofimov, Additive manufacturing of high entropy alloys: a practical review, J. Mater. Sci. Technol. 77 (2021) 131–162.

formation of submicrocellular grains in the CoCrFeMnNi HEA [302,325]. Additionally, a hierarchical microstructure consisting of columnar grains, submicron cellular structures, dislocation networks, and nanosized oxides were also achieved for CoCrFeMnNi printed-HEA [326]. The hierarchical microstructure of a CoCrFeNiMn high entropy alloy additively manufactured by PBF is shown in Fig. 1.25 [325]. Li et al. [327] developed an AlCoCrFeNiCu HEA with cemented carbide producing a tungsten carbide product via PBF. A fine W_2C /HEA dendritic structure was primarily formed, and the rapid solidification formed an

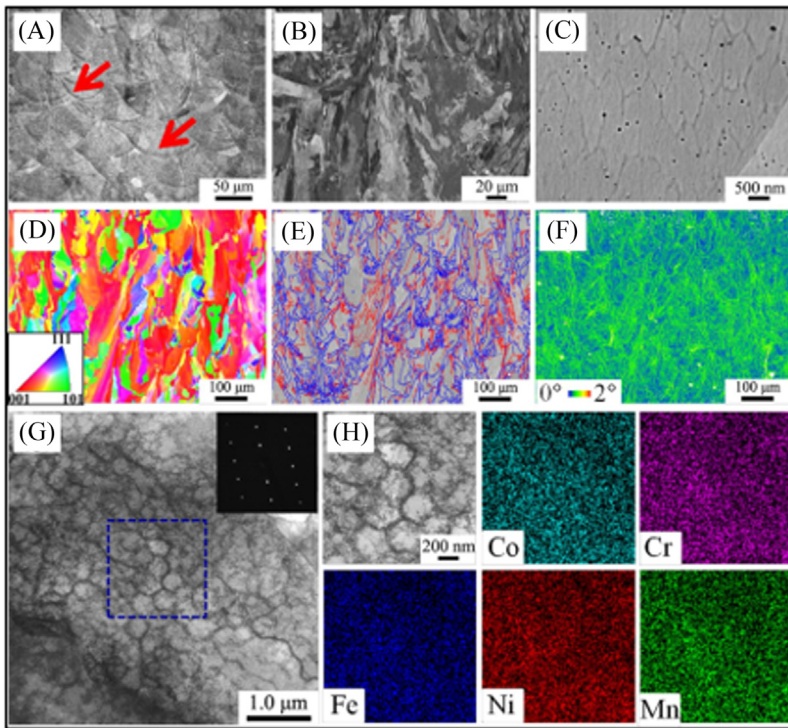


Figure 1.25 Microstructure characterization of CoCrFeNiMn HEA additively manufactured by SLM: (A) optical micrographs; (B) scanning electron micrograph; (C) scanning electron micrograph of the cellular structure; (D) electron backscattered diffraction results showing the inversion pole figure map; (E) electron backscattered diffraction results showing the image quality map with high angle grain boundaries (blue lines) and low angle grain boundaries (red lines) superimposed; (F) kernel average misorientation map; (G) bright-field scanning transmission electron microscopy image of the cellular structure with the corresponding selected area electron diffraction pattern; (H) bright-field scanning transmission electron microscopy image of the selected area illustrated by a square in (G) and the elemental distribution maps of the area.

Source: From Z.G. Zhu, et al., Hierarchical microstructure and strengthening mechanisms of a CoCrFeNiMn high entropy alloy additively manufactured by selective laser melting, *Scr. Mater.* 154 (2018) 20–24.

interdendritic fcc phase. Precipitation and coarsening of faceted WC from the W_2C dendrite occurred due to repeated heating by iterative laser scanning.

The multipowder delivery system enables DED to print hierarchical and functionally graded materials [328]. The lower scanning speed in DED results in a lower crystal growth rate and cooling rate compared to PBF, and larger grain sizes in DED-printed HEA products are achieved. The rapid heating and cooling rates cause high residual stresses in DED- and PBF-printed products, affecting the microstructure, thus, governing their macroscopic mechanical properties. Electron beam melting (EBM) products can achieve minor residual stresses due to the preheating for powder layers, reducing cracks in the final products [300,329]. The EBM-printed $Co_{1.5}CrFeNi_{1.5}Ti_{0.5}Mo_{0.1}$ HEA was compared to PBF-printed counterparts [330]. Single cubic and fcc phases were present in both products, but Ni_3Ti brittle intermetallic compounds precipitated only in the EBM-printed one. Wang et al. [223] performed the process optimization of EBM for $CoCrFeMnNi$ using a gas-atomized HEA powder and columnar grains with intragranular cellular grains along the build direction with a strong $\langle 100 \rangle$ texture were achieved. Fig. 1.26 [331] shows the microstructure of EBM built $CoCrFeNiMn_{0.18}Ti$.

1.5.5 Mechanical properties of 3D-printed HEAs

The wide ranges of strength and elongation of 3D-printed HEAs are more dependent on the compositions than on the process parameters in each printing process [215]. Table 1.6 [216] summarizes the tensile properties of HEAs fabricated by different AM techniques. Tensile yield strengths from 194 MPa for an fcc-based microstructure to 773 MPa for a bcc-based microstructure are exhibited for 3D-printed HEAs [215]. The PBF-printed HEAs show better tensile properties than EBM-printed ones due to refined microstructure and uniform composition without intermetallic compounds [215]. Exceptional elongations with improved strength of printed are achieved for $AlCoCrFeNi$, and $CoCrFeMnNi$ HEA indicates a strength–ductility synergy [215]. The addition of carbon improved the tensile strength of PBF-printed $CoCrFeNi$ HEA [341], attributed to the combination of dislocation networks strengthening and nanosized carbides strengthening [342].

The tensile behavior of the PBF-built $(CoCrFeMnNi)_{99}C_1$ HEA, and its comparison with other alloys is shown in Fig. 1.27 [313]. The influence of the volumetric energy density on the relative densities of PBF-printed $CoCrFeMnNi$ and $AlCoCrFeMnNi$ HEA is shown in Fig. 1.28 [216]. A Co-free $AlCrCuFeNi$ HEA was developed by Luo et al. [272]. The fracture strength and ductility perpendicular to the build direction were higher than those aligned in parallel due to the strong $\langle 100 \rangle$ texture in the perpendicular direction and precipitation of Cu-rich precipitates at the grain boundaries. The optimization of energy density resulted in the increase in the relative densities, change of crystallographic directions, reduction of grain sizes, and improvement on mechanical properties of printed HEAs [264,272,302,306,325,341,343].

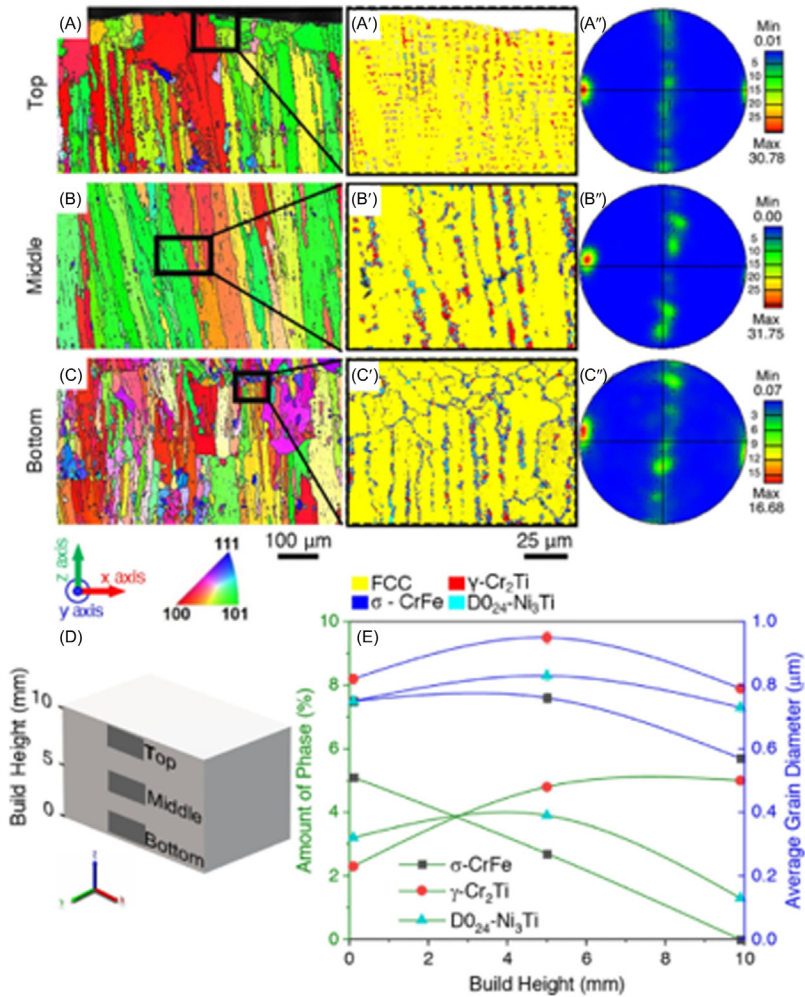


Figure 1.26 Microstructure of EBM built CoCrFeNiMn_{0.18}Ti. (A, B, C) Electron-backscattered diffraction orientation maps of fcc solid solution (inverse pole figure maps). (A', B', C') Phase distribution maps. (A'', B'', C'') Pole figures with texture index derived from electron-backscattered diffraction data. (D) Schematic illustration of electron-backscattered diffraction locations. (E) Existing phase fractions and average grain diameters of respective phases according to the build height and analysis locations.
Source: From M. Cagirci, et al., Additive manufacturing of high-entropy alloys by thermophysical calculations and in situ alloying, J. Mater. Sci. Technol. 94 (2021) 53–66.

Furthermore, heat treatments can improve the mechanical properties of 3D printed HEAs by removing various defects and releasing the residual stress existing in DED- and PBF-printed HEAs. Recrystallization occurs during annealing, reducing or eliminating the reduced residual stress. The effect of heat treatments of hot

Table 1.6 Tensile properties of HEAs fabricated by different AM techniques.

Composition	AM route	Phase structure	Mechanical properties				References
			T [K]	σ_y [MPa]	UTS [MPa]	Strain [%]	
CoCrNi	DED	fcc	RT	490	790	57	[271]
CoCrFeNi	Ink-extrusion	fcc	RT	250	598	33.8	[332]
			130	388	864	37	
CoCrFeMnNi	LAM	fcc	RT	352	540	26	[263]
			77	564	891	36	
CoCrFeMnNi	LMD	fcc	RT	290	535	55	[264]
			200	304	610	73	
			77	402	878	95	
CoCrFeMnNi	LAAM	fcc	RT	518	660	19.8	[266]
			143	710	850	40.2	
CoCrFeMnNi	LAM	fcc	RT	346	566	27	[267]
CoCrFeMnNi	DED	fcc	RT	424	651.3	47.9	[269]
CoCrFeNiNb _{0.1}	DED	fcc + Laves	RT	~ 400	~ 650	~ 55	[270]
CoCrFeNiMo _{0.2}	LMD	fcc	RT	~ 300	560	51	[333]
			77	~ 500	928	32	
CoCrFeNi	PBF	fcc	RT	600	745	32	[297]
CoCrFeMnNi	PBF	fcc	RT	519	601	34	[301]
CoCrFeMnNi	PBF	fcc	RT	510	~ 610	36	[296]
CoCrFeNiC _{0.05}	PBF	fcc	RT	708	872	~ 15	[334]
CoCrFeNiC _{0.05} @	PBF	fcc	RT	787	950	~ 10.5	
CoCrFeNi-(1.8 at.%)N	PBF	fcc	RT	650	853	34	[335]
CoCrFeMnNi-(1 at.%)C	PBF	fcc	RT	829	989	24.3	[336]
	PBF	fcc + TiCo ₂ + MoFe ₂		773	1178	25.8	[300]

(Continued)

Table 1.6 (Continued)

Composition	AM route	Phase structure	Mechanical properties				References
			T [K]	σ_y [MPa]	UTS [MPa]	Strain [%]	
Co_{1.5}CrFeNi_{1.5}Ti_{0.5}Mo_{0.1}	PBF + ST [#]	fcc	RT	897.5	1291	26.7	[337]
	EBM			743.4	832.2	40	
	EBM + ST			759	1139	35	
CoCrFeNi	PBF	fcc	RT	581.9	707.9	20	[338]
Al_xCoCrFeNi^{\$}	DED	fcc	RT	200	~ 1300	100	
$x = 0.3$		fcc, bcc		400	~ 1375	50	[276]
$x = 0.6$		bcc		1400	~ 2100	25	
$x = 0.85$							
AlCoCrFeNi_{2.1}	DED	bcc + L1 ₂	RT	678	1495	16	[275]
Al_{0.5}CoCrFeNi	PBF	fcc + bcc	RT	609	878	18	[308]
Al_{0.3}CoCrFeNi	LENS	fcc + L1 ₂	RT	410	~ 525	28	[274]
Al_{0.5}CoCrFeNi	PBF	fcc	RT	579	721	22	[307]
Al_{0.3}CoCrFeNi	PBF	fcc	RT	730	896	29	[309]
AlCrCuFeNi₃	PBF	fcc + bcc (B2)	RT	~ 850	957	14.3	[314]
AlCrFeNiV^{\$}	PBF	fcc + L1 ₂	RT	651	1057	30.3	[315]
AlCoCrFeNi	EBM	bcc + fcc	RT	769	107.5	1.2	[339]
CoCrFeMnNi	EBM	fcc	RT	205	497	63	[222]
Fe₄₀Mn₂₀Co₂₀Cr₁₅Si₅	PBF	ϵ (hcp) + γ (fcc)	RT	~ 530	1100	30	[322]
Fe_{49.5}Mn₃₀Co₁₀Cr₁₀Co_{0.5}	PBF	fcc	RT	710	1000	28	[340]
Ni₆Cr₄WFe₉Ti	PBF	fcc	RT	710	983	12.9	[321]

LAM, Laser additive manufacturing; LMD, laser metal deposition; LAAM, laser aided additive manufacturing; LENS, laser engineered net shaping; [@] Annealed at 1073K for 0.5 h; [#]ST, solution treated at 1393K for 3 h followed by water quenching; ^{\$} tensile properties are true vales.

Source: From A. Ostovari Moghaddam, N. A. Shaburova, M.N. Samodurova, A. Abdollahzadeh, E.A. Trofimov, Additive manufacturing of high entropy alloys: a practical review, J. Mater. Sci. Technol. 77 (2021) 131–162.

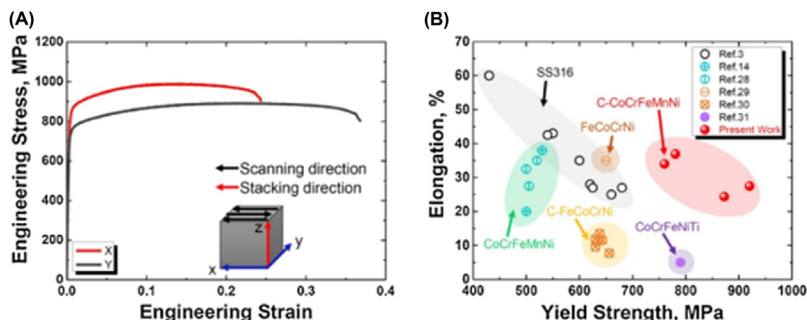


Figure 1.27 (A) Tensile behavior of the SLM-built $(\text{CoCrFeMnNi})_{99}\text{C}_1$ HEA along the x and y directions. (B) The yield strength vs elongation values for $(\text{CoCrFeMnNi})_{99}\text{C}_1$ and some alloys as shown in [342].

Source: From J.G. Kim, et al., Nano-scale solute heterogeneities in the ultrastrong selectively laser melted carbon-doped CoCrFeMnNi alloy, *Mater. Sci. Eng. A* 773 (2020), 138726.

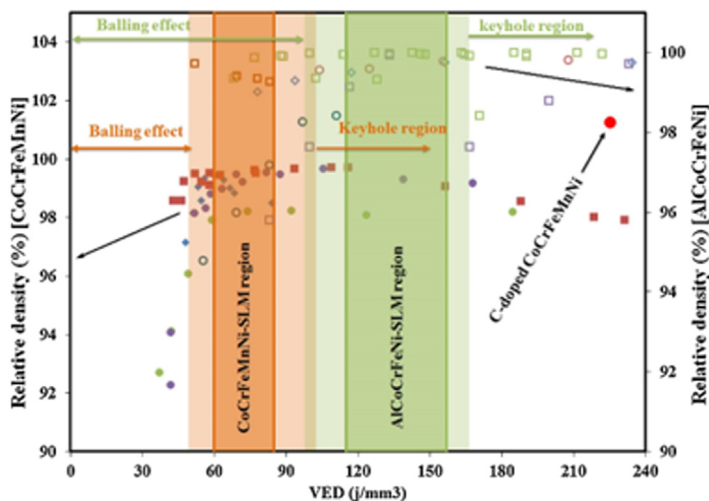


Figure 1.28 Relative densities of PBF-printed CoCrFeMnNi and AlCoCrFeMnNi HEA as a function of volumetric energy density (VED). Solid and open symbols represent the density of CoCrFeMnNi and AlCoCrFeMnNi HEA, respectively. The VED ranges corresponding to balling effect, keyhole porosity, and sound printing are labeled. The transparent colors indicate transition regions.

Source: From S. Luo, et al., Selective laser melting of an equiatomic AlCrCuFeNi high-entropy alloy: processability, non-equilibrium microstructure and mechanical behavior, *J. Alloy. Compd.* 771 (2019), 387–397.

density. Powder- or wire-based laser/electron beam melting process on the other hand pose a challenge for the mostly very brittle hard magnetic materials, which are prone to cracking and delamination due to the fast cooling rates in these techniques. For the wire-based techniques, it is often impossible to get a wire starting material for very brittle magnetic alloys. The following subsections will deal with the most interesting and promising magnetic materials manufactured with different methods and the challenges inherently tied to them.

1.6.2 Nd-Fe-B

Nd-Fe-B is a widely used and currently the strongest hard magnetic material available for the application in a moderate temperature range (RT up to 180°C). Nd-Fe-B crystallizes in a complex tetragonal crystal structure as illustrated in Fig. 1.30 [346]. It is rather brittle, making the material prone to cracking during additive manufacturing where high heating and cooling rates are part of the process. An additional challenge in the additive manufacturing of this alloy is the decomposition of the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase into an Nd-rich and a Fe-rich phase at higher temperatures, which is detrimental to the magnetic properties [346].

When using SDS additive manufacturing strategies, one does not face these challenges since in these techniques mixture of an Nd-Fe-B powder with a polymer, usually, a filament is formed, which is then used in a polymer printed. The mixture is only exposed to temperatures up to the melting point of the polymer, and the fabricated parts can then be used as printed. Another alternative relies on a two-step debinding (a chemical followed by a thermal one), where the polymer can be removed by a solvent dissolution followed by a pyrolysis step with the resulting green body of Nd-Fe-B to be sintered [347]. A disadvantage of these techniques is the resulting lower relative density of the printed parts

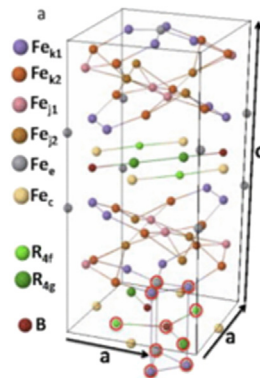


Figure 1.30 Crystal structure of Nd-Fe-B in the Wyckoff notation, R = Nd.

Source: From A. Alam, M. Khan, R.W. McCallum, D.D. Johnson, Site-preference and valency for rare-earth sites in (R-Ce) 2 Fe 14 B magnets, Appl. Phys. Lett. 102 (2013) 042402.

and therefore reduced magnetic properties. Several papers have been published on the additive manufacturing of Nd-Fe-B [348–352], Mn-Al-C [353–355], and Sr ferrites [353,356]. Different polymers can be used for the production of the filament material, which has an influence on the final mechanic and magnetic properties as well as on the temperature range, the final magnetic parts can be used [351].

Big Area Additive Manufacturing (BAAM), in comparison, uses the polymer-magnet powder mixture in pellet form to produce Nd-Fe-B part of considerable magnetic properties [coercivity (H_c) = 708.2 kA/m and remanence (B_r) = 0.58 T] [350,357,358]. In comparison to FFF, BAAM is a technique perfectly suitable to produce larger parts layer by layer with a big nozzle (Fig. 1.31A) by depositing the molten polymer-magnet mixture (Fig. 1.31B). The advantage of this method is the high structural flexibility coupled with a low eddy current loss in the printed parts due to low conductivity of the polymer. The mechanical properties are hereby largely defined by the applied polymer.

The issue of low density in hybrid polymer/metal printed and subsequently sintered parts as well as in direct laser sintered parts, however, can be resolved when using different infiltration techniques. The porous structures of sintered parts are especially suitable to be infiltrated by low-melting point eutectic Nd-rich alloys. The infiltration agent is wrapped around the printed part, which is then heat-treated at elevated temperatures. The molten eutectic alloy fills the pores in a first step and in a second step by a grain boundary diffusion process, the alloy then diffuses into the grain boundaries of the Nd-Fe-B parts. In the end, this increases density, mechanical properties and coercivity [359,360].

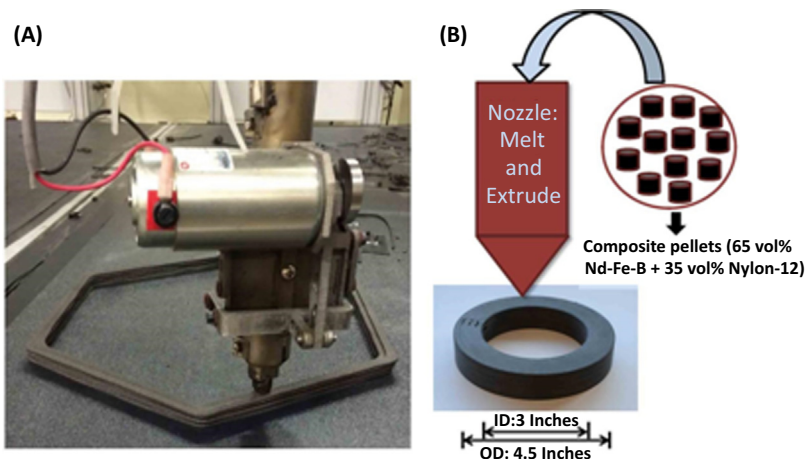


Figure 1.31 Big Area Additive Manufacturing of Nd-Fe-B. An Nd-Fe-B/ Nylon-12 composite is formed (A) which is then used in BAAM to print stable, 3D-printed magnets of large size (B).

Source: From L. Li, et al., Big area additive manufacturing of high performance bonded NdFeB magnets, Sci. Rep. 6 (2016) 36212.

A similar approach is to use selective melting of one material in the LPBF process. Here, a powder mixture of the main material (i.e., Nd-Fe-B) and the infiltration material (i.e., a low-melting point, eutectic NdPrCoCu alloy) is prepared and the use of low energy densities can cause a selective melting of the lower melting point Nd-rich alloy in the process. This results in a different consolidation mechanism, melting the infiltration agent while leaving the higher melting point Nd-Fe-B powder unmolten (see Fig. 1.32). Here, an infiltration of the grain boundaries in consolidated Nd-Fe-B powder by the Nd-rich alloy can be observed, which results in improved magnetic properties and greater temperature stability of coercivity [361].

A necessary step to overcome the obstacle of cracking and low relative densities in powder bed fusion techniques are advanced parameter studies, which can include in situ preheating of each layer, double-melting, deburring, and examinations of the melt pool behavior with different parameters [362–365]. Usually, densities of the printed parts are below 95% and the processing window here is still very narrow. Melt Pool Studies and models can help to correlate printing parameters with the resulting magnetic properties [362,364]. In a melt pool study, single lines of the material are printed with a defined layer height. The diameter and height of the cross-section (h and L in Fig. 1.33A) and the contact angles η_1 and η_2 (Fig. 1.33B) are measured to determine the adhesion of the melt pool to the surface. Top views of the melt tracks are made to distinguish parameters resulting in stability (Fig. 1.33C) from parameters resulting in unstable melt tracks (Fig. 1.33D). With these parameters, the stability of the melt pool can be determined [362,366].

As state of the art, a coercivity (H_c) of 921 kA/m with a remanence (B_r) of 0.63 T, and an energy product ($BH_{\max}$) of 63 kJ/m³ can be achieved without further postprocessing [365]. All studies above show the steep challenge in the additive manufacturing of this material with powder bed melting techniques to achieve highly dense and crack free samples. Promising results have been achieved at our TU Graz when using

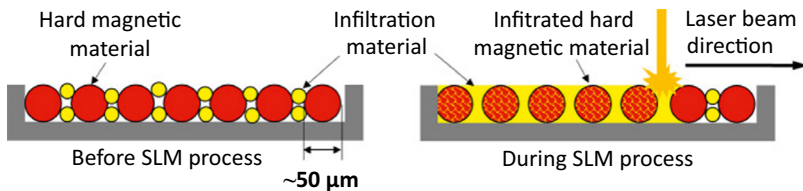


Figure 1.32 In situ grain boundary infiltration of Nd-Fe-B with a lower melting eutectic alloy using the LPBF technique. In the printing process, only the lower melting component of the powder mixtures is molten. The molten alloy acts both as a binder and an infiltration agent, diffusing into the grain boundaries of the Nd-Fe-B grains. The grain boundary infiltration of the grains results in an increased coercivity due to a decoupling of the magnetic moments of neighboring grains.

Source: From A.S. Volegov, et al., Additive manufacturing of heavy rare earth free high-coercivity permanent magnets, *Acta Mater.* 188 (2020) 733–739.

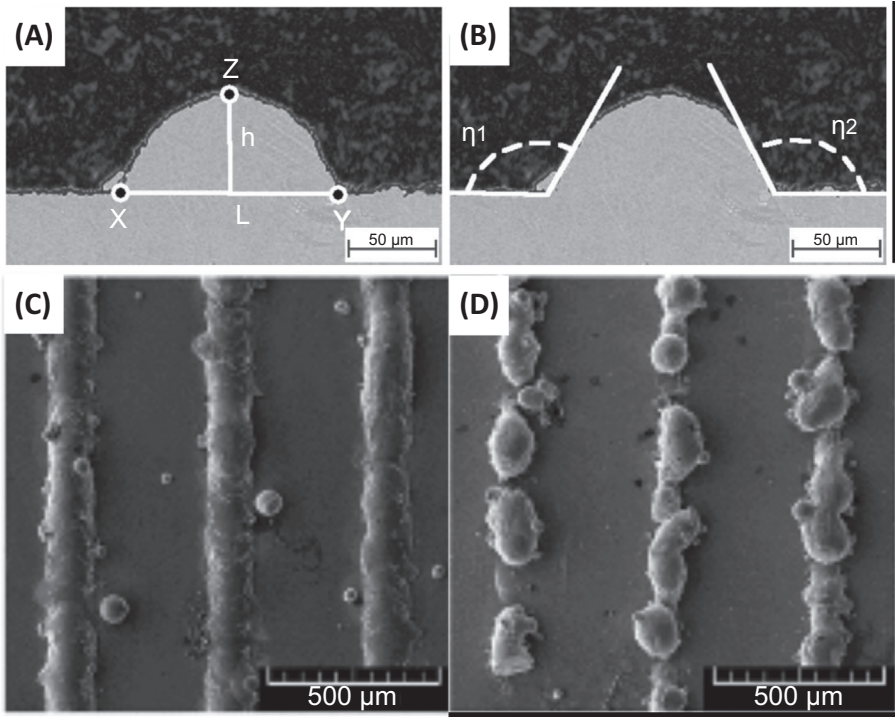


Figure 1.33 Melt Pool Analysis of different parameters in Nd-Fe-B (example images). Relevant parameters to be determined are (A) diameter and height of the cross-section of the single-track, (B) wetting angle of the single track. The wetting angle should be above 90° for a stable melt pool: (C) stable single line tracks and (D) unstable single line tracks (balling).
 Source: From M. Skalon, et al. Influence of melt-pool stability in 3D printing of NdFeB magnets on density and magnetic properties, Materials (Basel) 13 (2020) 139.

a large spot size in combination with small hatch spacing. This approach results in an overlap of the laser spots, which is assumed to decrease the cooling rate through reheating of the previous melt lines (see Fig. 1.34). Further parameter studies have to be performed to enable more stable, dense and crack-free samples.

1.6.3 Fe-Co-based magnetic alloys

Fe-Co-based magnetic alloys are characterized by an alloy system of Fe and Co in combination with other alloying elements like Al, Ni or Cr that exists in a b.c.c. α -phase at temperatures above 1000°C . This α -phase decomposes in a hard-magnetic, Fe-Co-rich, b.c.c. α_1 -phase and a weak-magnetic α_2 matrix (b.c.c. or intermetallic phase) [367–370]. Prominent examples are the commercially available Al-Ni-Co and Fe-Cr-Co alloys. These alloy systems have the advantage of

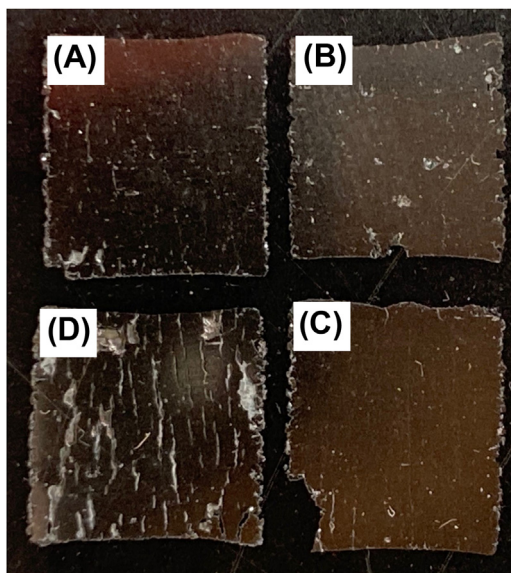


Figure 1.34 Nd-Fe-B samples manufacture via the LPBF process with different hatch spacings. Common parameter: laser power = 100 W, laser spot size = 120 μm , layer height = 40 μm , laser speed = 521 mm/s. Changing parameter: hatch spacing: (A) 30, (B) 40, (C) 50, and (D) 140 μm . An overlap of the laser spot seemed to influence the occurrence of cracks in the samples.

being free from rare-earth metals (RE); however, the hard magnetic properties are considerably weaker than in Nd-Fe-B.

Since rare-earth metals are a critical resource and the demand for permanent magnetic materials steadily increases due to the ongoing electrification, additively manufactured alternatives to RE containing magnetic materials like Fe-Co-based alloys are becoming more popular in areas, where the high magnetic properties per volume inheritance to Nd-Fe-B or Sm-Co are not explicitly needed. These alloys also have the advantage of being operable at temperatures up to 400°C and theoretical calculations have shown, that the magnetic properties can be enhanced by a factor of $2\text{--}3 \times$ with a properly improved microstructure [367]. Additive manufacturing and its wide range of process conditions might pave the way to achieve this goal.

White et al. [371,372] have worked on the production of Al-Ni-Co alloys with three different AM methods—laser engineered net shaping (LENS), directed energy deposition (DED), and electron-beam powder bed fusion (EB-PBF)—achieving magnetic properties for AlNiCo 9 close to the properties of comparable as-cast AlNiCo alloys (around 170 kA/m); the sample is depicted in Fig. 1.35. In a detailed study on the influence of printing parameters on the final magnetic microstructural properties of two AlNiCo alloys with varying Co-content, the authors identified the DED method as the more suitable in comparison to the EB-PBF method. This is

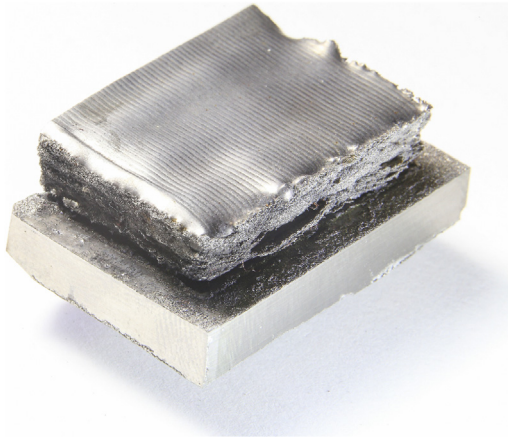


Figure 1.35 DED fabricated Al-Ni-Co sample.

Source: From E. White, et al., Processing of alnico magnets by additive manufacturing, Appl. Sci. 9 (2019) 4843.

most likely due to the evaporation of Al and alloyed Cu, which is facilitated by the vacuum atmosphere of the process.

1.6.4 Additive manufacturing of soft magnetic materials

The additive manufacturing of soft magnetic alloys often poses fewer challenges regarding the process itself due to the generally better machinability of soft magnetic alloys in comparison to most hard magnetic alloys. Although there are also problems regarding machinability arising when considering soft magnetic alloys like pure Fe-Co, even these alloys are less brittle when compared to their hard-magnetic counterparts like Nd-Fe-B. This puts the soft-magnetic alloys in a kind of a sweet spot for additive manufacturing: The machinability of these alloys is too poor to produce more complex structures with conventional methods, nevertheless they are relatively easy to print in comparison to mentioned above hard magnetic alloys.

Regarding the microstructural and magnetic properties of 3D-printed, soft-magnetic parts, different results can be achieved by choosing different printing parameters. To obtain optimal soft magnetic properties, coarse grain size and a well-aligned crystallographic texture, preferably with the easy axis of the material aligned alongside the magnetization direction in the application, is desired.

Here, certain defects known to occur in various AM processes (state of disorder, oxide inclusions) can negatively affect the soft magnetic properties. The most common materials, currently worked in using AM are Fe-Ni, Fe-Co, or Fe-Si based [373]. In near equiatomic Fe-Co, an ordering from an α -b.c.c. phase to a B2-ordered phase occurs during cooling, which is detrimental to the workability of those alloys. In a study by Kustas et al. [374], thermal measurements were performed to determine the cooling rates using the LENS method and interlayer

intervals were introduced to decrease the temperature of the built part and to subsequently increase the cooling rate. With higher cooling rates, lower states of order could be found. High cooling rates on the other hand also resulted in fine-grain structures with low degrees of texture, which in turn result in higher coercivities and a lower saturation magnetization when compared to annealed samples and comparable values found in the literature.

To produce a soft magnetic alloy in situ, a Fe-Si steel powder can be coated with Ni on the surface to obtain Fe-Ni-Si during printing. The printed parts exhibit a proportional dependency of density and an indirect proportional dependency of occurrence of cracks to scanning speed as shown in a study by Kang et al. [375]. With higher scanning speed, a certain fraction of f.c.c.-(Fe,Ni) phase, as well as lower grain size, can be observed, which results in worse weak magnetic properties. In samples with low scanning speed, only the b.c.c.-(Fe,Ni) phase is formed, which is preferential for the magnetic properties.

When using LENS to produce a Fe-30%Ni alloy a general trend can be shown that with increasing scanning speed, a higher percentage of martensitic b.c.c. phase is present with pure b.c.c. at highest applied speeds. At lower scanning speeds, only f.c.c. phase is present. The formation of b.c.c. phase was attributed to stress-induced martensite formation due to higher cooling rates in a study done by Mikler et al. [376]. Good soft magnetic properties (H_c 1.5–2.3 kA/m) with a higher saturation magnetization (M_s 165 emu/g) can be achieved for samples higher in b.c.c. phase. This can be attributed to the greater magnetic moment of the b.c.c. phase.

Fe-30%Ni can be also produced via in situ alloying of pure Fe and Ni powders in the LPBF method. Due to the much higher cooling rates in LPBF, only b.c.c.-(Fe,Ni) phase can be observed in the examined 3D printed parts as shown in a study by Zhang et al. [377]. The higher cooling rates in the LPBF process also usually result in a finer grain structure, which can be disadvantageous to the soft magnetic properties.

In some applications, it may be required to have a part with a gradual change in magnetic properties. When such properties are necessary, printing structures with an elemental gradient might be a very suitable approach. The LENS method is perfectly capable of producing such a part out of a material where the magnetic properties change in the desired way with the chemical composition. Both Fe-Co and Fe-Ni can be suitable materials for this approach as shown in a study by Chaudhary et al. [378]. In the Fe-Ni system, the share in b.c.c. phase increases with increasing Fe content but remains below 5%, which can be attributed to the high cooling rate, consistent with what Mikler et al. [376] have found in their study. For the Fe-Co system, only b.c.c. phase can be found. For both alloys, saturation magnetization increases as expected with higher Fe content and coercivity peaks around the equiatomic region. Overall, magnetic properties are comparable to those of conventionally produced parts. With increasing Fe content, Curie temperature increases for the Fe-Co system and decreases for the Fe-Ni system. Grain sizes are in the range of 50 μm and no significant texture was found in the samples.

Considering the development of a preferential texture in magnetic materials, the LPBF method is known to produce different textures on certain occasions [379]. If certain textures are required, even the targeted design of a material with a defined texture

is possible [380]. In two consecutive studies, Garibaldi et al. [381,382] investigate LPBF manufactured Fe-Si samples considering the influence of the printing parameters and subsequent postheat treatment on the microstructure, ordering, texture, and magnetic properties. In both studies, a strong [001] texture in the building direction can be observed. Additionally, the best magnetic properties can be observed with medium energy inputs and after heat treatments at 700°C. This effect is attributed to the formation of a preferential texture at medium energy inputs, as well as stress relief and grain growth at higher annealing temperatures. Upon annealing, samples show ordering in the XRD measurements, which is not present in the print samples, where the ordering is most likely prevented by the high cooling rates.

1.7 In situ alloying

1.7.1 Introduction

The production of novel materials by additive manufacturing is highly reliant on feedstock. The difficulties range from feedstock production (wire drawing or alloy atomization) to high prices depending on alloy composition—elemental reactivity and price, protective atmosphere, special handling. In other words, the prices of prealloyed powders are sometimes practically prohibitive. Especially for powder-based techniques, a promising alternative is the use of in situ alloying where instead of using prealloyed feedstocks, powder mixtures are prepared to be printed by powder bed (such as laser or electron beam powder bed fusion techniques, LPBF and EBM, respectively) or direct deposition methods (DED). These mixtures can comprise elemental powders or simpler, two-element alloys (e.g., commercial Co-Cr) and are in most cases easier available than the prealloyed powder of the desired material. Mainly applied in powder bed fusion and direct energy deposition techniques, the use of powder mixtures poses some challenges, which will be discussed briefly in the following subsections. For a more detailed analysis of this technique, the reader is referred to check the following references [382–384].

1.7.2 Powder quality and mixing

The first challenge in using powder mixtures is the quality of each powder. Optimal powders for both powder bed and direct deposition-based techniques are spherical to enable a good flowability with particle size distribution (PSD) roughly in the range of 20–45 μm , although the quality of these properties is more relevant in the powder bed techniques [385]. For powder mixtures especially in powder bed fusion, in the most optimal case, the applied elemental powders have similar, narrow particle size distribution to achieve both a good mixing and a dense powder bed as well as to avoid agglomeration and separation during the printing process. Best properties can be achieved when each of the component powders of the mixture is perfectly spherical. As shown by Knieps et al. [383], the powder morphology can have a huge influence on the properties of the final part. This is consistent with the research on in situ

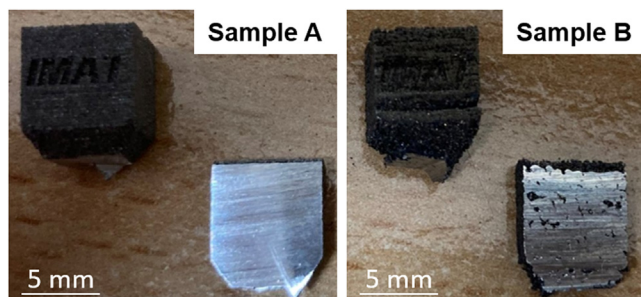


Figure 1.36 Two in situ alloyed samples with the same nominal composition (Fe-30.5Cr-15Co-1.5Mo) with the following parameter: spot size: 50 μm , hatch spacing: 60 μm , layer height: 25 μm , energy density: 280 J/cm^2 ; sample A: printed from a powder mixture composed of spherical powders only; sample B: printed from a powder mixture composed of partially spherical and partially aspherical powders. Powders used for these samples can be seen in Fig. 1.37.

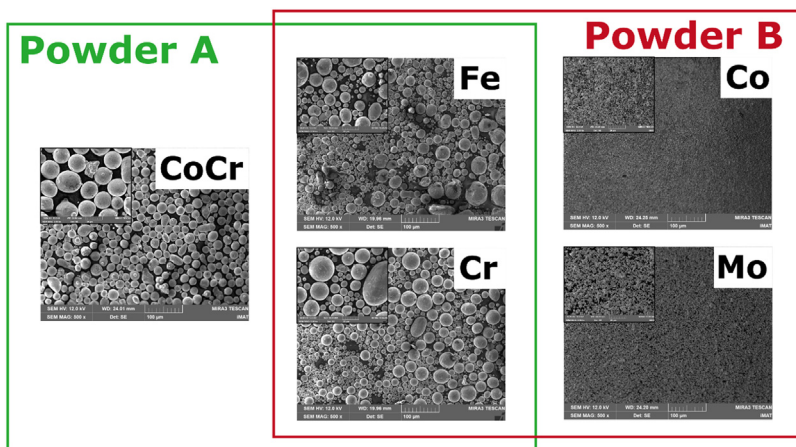


Figure 1.37 Powder mixtures were used to print samples A and B described in Fig. 1.1. Powder A: Fe from TLS Technik Germany, PSD 20–63 μm ; Cr from TLS Technik Germany, PSD 15–53 μm ; Co-29Cr-6Mo from Oerlikon GmbH, PSD 15–45 μm . Powder B: Fe and Cr as in powder A, Co from US-Nano, mean grain size 10 μm ; Mo from US-Nano, mean grain size 800 nm. Particle size descriptions as indicated by the manufacturer.

alloying as done at TU Graz, as can be seen in Figs. 1.36 and 1.37. The former figure shows two samples of the same nominal composition printed with the same set of process parameters (spot size: 50 μm , hatch spacing: 60 μm , layer height: 25 μm , energy density: 280 J/cm^2), however from a powder mixture with different morphologies of its components. In this example, a Fe-30.5Cr-15Co-1.5Mo (wt.%) alloy, powder mixture A is composed of Fe and Cr powder from TLS Technik GmbH, Germany and Co-29Cr-6Mo (wt.%) alloy from Oerlikon GmbH. Powder mixture B

was prepared from Fe and Cr from TLS Technik and Co and Mo powder from US-Nano (Fig. 1.37). As can be seen in these images, the Fe and Cr powders hereby were of good, the Co-29Cr-6Mo powder of very good and the Co and Mo powders of very poor quality regarding sphericity. The particle size distribution of the components was similar for powder mixture A and different for powder mixture B. Mo was purchased on purpose as a fine powder with a smaller mean diameter to enable better melting of the higher melting point element. This example shows that even exchanging two components in a powder mixture for in situ alloying, one may have a huge influence on the final properties of the printed part.

In many cases, the elemental powders might not have the same quality, especially when coming from different producers; this case requires special care when synthesizing the powder mixtures. Mixing time and mode of mixing can influence the quality of the final powder mixture and subsequently the printing process. A more detailed investigation on the mixing procedure of an in situ alloyed Ni-Ti alloy was done by Wang et al. [169] who observed agglomeration and reagglomeration at too short and too long mixing times. This suggests that when working with a new powder mixture, it might be necessary to do an investigation of the mixing behavior first, especially for component powders that strongly deviated from the perfectly spherical morphology. Additional process tools like stainless steel balls added to the powder mixture before mixing can also help facilitate proper mixing and prevent agglomerations in the process.

Another means to overcome improper mixing and desegregation in the printing process is to use a technique called satelliting, requiring powders of different particle size distributions. Here, one component purposefully consists of smaller particles that are attached to the larger ones of the other component via a binder or diffusion bonding. Simonelli et al. [386] made use of this technique by covering larger titanium particles with smaller Al and V particles, aiming to produce Ti-6Al-4V parts and bonded via polyvinylalcohol (PVA). The authors of this study compared this technique with conventional mixing and were able to show an improved chemical homogeneity in the printed parts. However, the shape of the Al and V powders used was not perfectly spherical, which might also have contributed to the inhomogeneity in the printed parts produced with the conventionally mixed powder due to a different flowability. Another disadvantage of this technique might be a decrease overall in flowability in comparison to the initial powder, given the case that all component powders were perfectly spherical. For powder mixtures where only the main powder is spherical—as in this case, Ti—a significant decrease in flowability when using satelliting might not occur, which would make this method perfectly suitable to increase the quality of these kinds of powder mixtures.

1.7.3 The temperature of melting, energy input, and homogeneity

Another issue to be thought of when considering the use of powder mixtures is a difference in energy input needed to melt different powders. It can be either due to

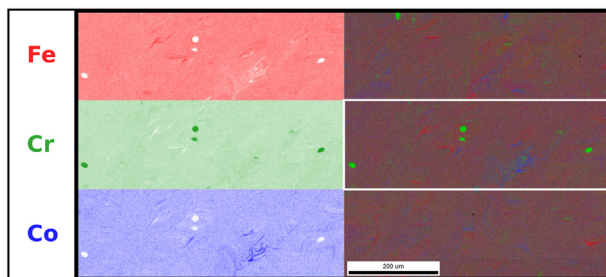


Figure 1.38 Energy dispersive X-ray spectroscopy map showing the elemental distribution in in situ alloyed Fe-30.5Cr-15Co-1.5Mo. The images on the left show the maps of the three main component elements taken from the region marked with a white box in the overlay image on the right. In an otherwise relatively homogeneous material, regions of unmolten Cr particles can be seen.

the different melting temperatures of the elemental components or related to the different reflectivity of the powder particles when applying laser-based AM techniques. For instance, aluminum alloys are known to require higher energy inputs in the printing process relative to their low melting temperature [387]. The aforementioned issues may lead to inhomogeneities in the in situ alloyed material or segregation of certain elemental components. That said, Fig. 1.38 illustrates the in situ alloying process of a magnetic Fe-30.5Cr-15Co alloy printed using the following parameters: laser power of 190 W, a spot size of 40 μm , hatch spacing of 105 μm , layer height of 25 μm , and energy density of 280 J/cm². Depending on the parameters chosen, Cr—which has a higher melting point—may remain partially unmolten in an otherwise relatively homogeneous Fe-30.5-xCr-15Co matrix, as seen in the white encircled region where green (Cr) particles are highlighted.

Fig. 1.39 reveals the influence of laser power (LP) and energy input (ED) on the element distribution of the in situ alloyed Fe-30.5Cr-15Co-1.5Mo printed part. For this case, common parameters such as layer height (25 μm), spot size (50 μm), hatch spacing (60 μm) were kept constant. This image exhibits how unmolten particles vanish by changing LP and ED; elemental homogeneity in the sample seems to increase with increasing energy density, while the laser power seems to have less influence. Huber et al. [388], who investigated the influence of printing parameters on the dissolution of high melting point elements in a lower melting point matrix by in situ alloying in LB-PBF, found a similar correlation. Dissolution of the high-melting-point elements increased with increasing energy density. However, contrary to the findings for in situ alloyed Fe-30.5Cr-15Co-1.5Mo, where lower laser powers seem to result in slightly less homogeneous samples, lower laser powers here seemed to facilitate the dissolution of the higher melting point elements.

The influence of hatch spacing on the alloy homogeneity is shown in Fig. 1.40 for the samples printed according to the following set: laser power of 190 W, energy input of 280 J/cm², layer height of 25 μm , a spot size of 50 μm . The hatch spacing is (A) 100 μm , (B) 200 μm , and (C) 300 μm . Here, different melt pool sizes, which can

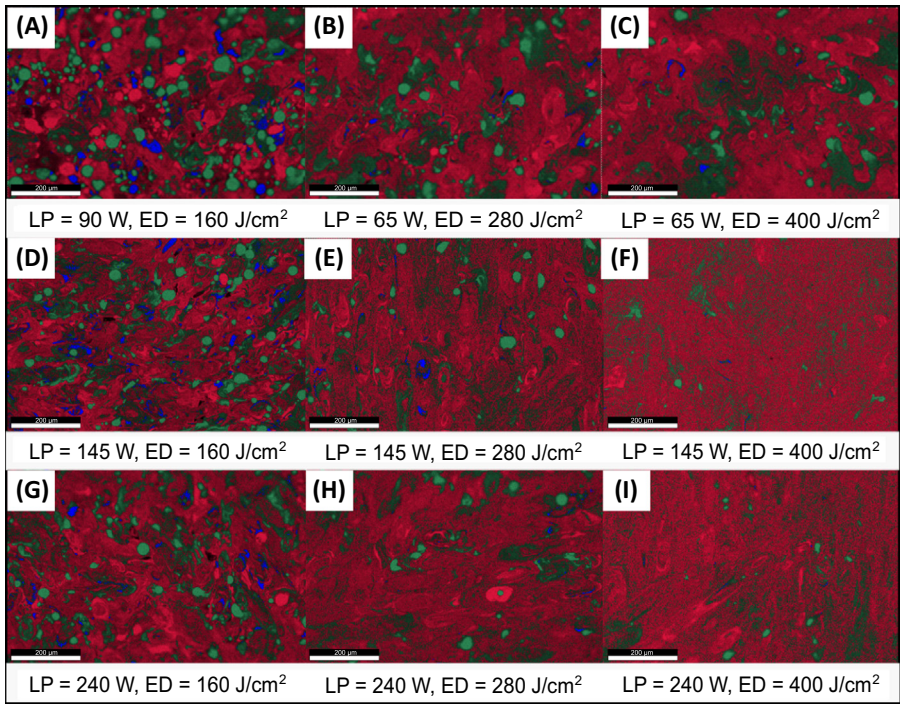


Figure 1.39 Elemental homogeneity in situ alloyed Fe-30.5Cr-15Co-1.5Mo printed with varying laser powers and energy densities.

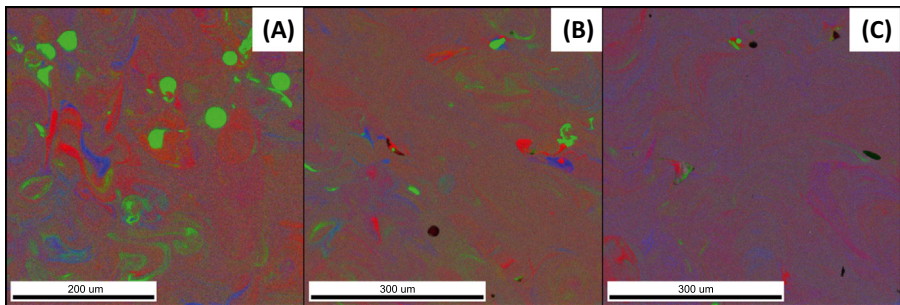


Figure 1.40 Elemental homogeneity in situ alloyed Fe-30.5Cr-15Co-1.5Mo printed with varying hatch spacings: (A) 100 μm , (B) 200 μm , and (C) 300 μm .

be achieved by a variation of the hatch spacing, are shown to have a strong influence on the homogeneity. A larger melt pool (larger hatch space) usually resulted in a more homogeneous material, as depicted by Fig. 1.5. These results are consistent with the findings of Wang et al. [169], showing that direct energy deposition

exhibiting a larger melt pool also results in a better homogeneity in the final printed parts in comparison to the other two methods observed, namely LPBF and EBM.

Inhomogeneous samples might not be inherently disadvantageous. In many cases, if stable and dense solid parts are achieved, homogenization treatments can be applied afterward to achieve the desired elemental homogeneity. Depending on the application of the produced part, the microstructural evolution during the treatment has to be carefully assessed once it can either lead to the desired microstructure or a detrimental one. For each novel alloy, these investigations are of utmost importance: by comparing temperature and time it is possible to determine the proper combination to achieve a homogeneous distribution of elemental components, as well as to observe the influence of these parameters on it. Such an investigation was performed on an in situ alloyed Fe-Cr-Co alloy (spot size of 50 μm , hatch spacing of 60 μm , layer height of 25 μm , and energy density of 280 J/cm²), exhibited in Fig. 1.41. In this alloy, a homogeneous α -phase is stable above 1000°C [389,390]. At 1000°C of annealing temperature, no sufficient homogenization could be achieved in 2 hours of

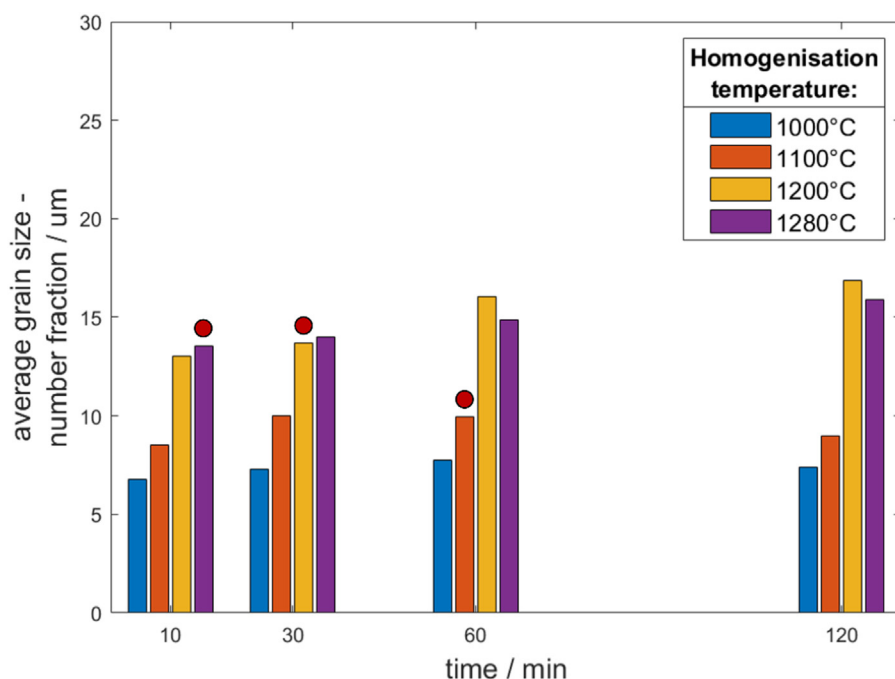


Figure 1.41 Evolution of average grain size distribution with homogenization time and temperature in an in-situ alloyed Fe-30.5Cr-15Co-1.5Mo alloy. The average grain size and homogenization was determined by electron backscattered diffraction analysis coupled with energy-dispersive X-ray spectroscopy. Scan Area of 800 × 650 μm , a voltage of 20 kV, beam intensity of 16 mA, and dwell time of 18Ms. The time at which homogenization could be achieved at each temperature is indicated with a red dot: 1280°C (10 min), 1200°C (30 min), and 1100°C (60 min).

annealing. Upon annealing, average grain size increases strongly with temperature and slightly with time. With higher temperatures, faster homogenization can be achieved, which is accompanied by larger grain growth.

1.8 AM of recycled Ti-64 powder

1.8.1 Introduction: why reuse the powder?

The economical success of powder bed-based metal additive manufacturing processes such as laser and electron powder bed fusion (L-PBF and E-PBF, respectively) and binder-based PBF is highly dependent on reusing the powder after each building cycle. While in binder-based processes, the unused powder is not exposed to energy input, in laser and even more so electron beam processes most of the powder is affected. This chapter provides a basic overview of side effects to consider when reusing powder by the example of the widely used titanium alloy Ti6Al4V Grade 5 (Ti64). To evaluate the influence of the reuse of powder, first, the impact on the powder itself is shown. Following, changes in build specimens and their mechanical properties are presented.

1.8.2 Influence on the powder

The main properties to characterize the suitability of powder for the L-PBF process are particle size distribution (PSD), sphericity flowability, and its bulk or tap density. For safe reuse of powder, these parameters must stay within the limits required for the L-PBF process. As seen in [Fig. 1.42](#), for reusing Ti64 12 times Meier et al. [\[391\]](#)

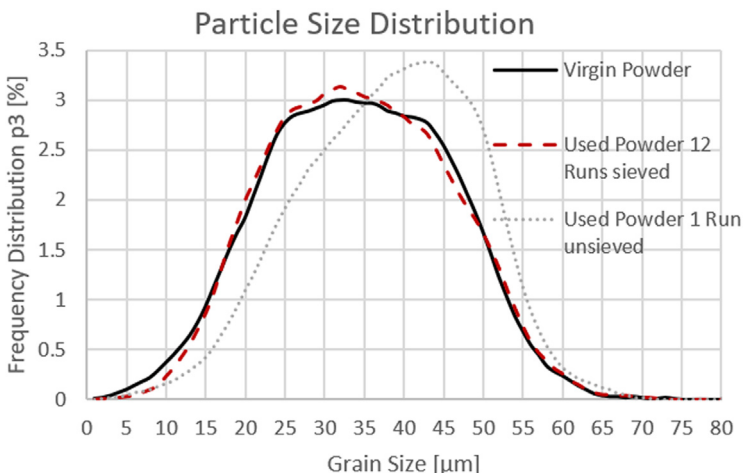


Figure 1.42 PSD of the virgin, once (not sieved) and 12 times reused (sieved) powder.

showed no significant shift of particle size distribution toward a larger mean diameter (D50) besides a stable maximum particle size. A minor reduction in fine proportion below 15 μm and a more defined peak at D50 was observed. For a singular use cycle, the PSD shifts toward larger diameters before sieving. Surprisingly, sphericity is found to be steady while flowability and bulk density increase.

The effects in Table 1.7—also found in the work of Quintana et al. [392] and Carrion et al. [393]—though additionally, a small shift in D50 occurs. Overall, this indicates the effectiveness of the sieving process concerning deformed and partially melted particles and no limitation for multiple reuses, although a regular evaluation of PSD is recommended.

Besides being a reactive material that forms a stable and even oxide layer at room atmosphere, titanium, and its alloys are prone to intake or even react with air components such as oxygen, hydrogen, and nitrogen especially at a higher temperature. Even though kept under argon atmosphere, during a building job, remaining oxygen can not be excluded. In addition, in most cases, powder handling is performed under the air atmosphere, which can lead to further pollution. Apart from the intake of gaseous components, alloying elements such as aluminum have both lower melt points as well as steam pressure and therefore might evaporate. An example of changes in chemical composition can be found in Table 1.8.

Table 1.7 PSD, sphericity, flowability, and bulk density of virgin and used powder.

Powder	D10 [μm]	D50 [μm]	D90 [μm]	w/h	SPHT13	Flowability [s/50 g]	Bulk density [g/cm ³]
Virgin	18.8	33.7	48.7	0.895	0.94	—	2.34
Used (1 job, unsieved)	21.8	37.4	50.3	0.894	0.94		
Used (12 jobs, sieved)	18.4	32.6	48	0.898	0.94	27	2.43

Table 1.8 Chemical composition of virgin and used powder.

Powder	Al [wt.%]	O [wt.%]	N [wt.%]	Ar [ppm]	H [ppm]
Virgin	6.13	0.16	0.0281	1.1	21
Used (1 run, unsieved)	6.23	0.16	0.0323	1.2	20
Used (12 runs, sieved)	6.40	0.18	0.0358	1	23
Limits Grade 5 (ASTM B348)	5.50–6.75	max 0.20	max 0.50		125
Error	± 0.332	± 0.006	± 0.00444		

Table 1.9 Chemical composition of virgin and used powder using a preheated build platform.

Powder	Al [wt. %]	O [wt. %]	N [wt. %]	Ar [ppm]	H [ppm]
Virgin	6.380	0.090	0.008		20
Used—220°C Preheating	6.050	0.120	0.017		20
Used—550°C Preheating	6.110	0.330	0.0150		168
Limits Grade 5 (ASTM B348)	5.50–6.75	max 0.20	max 0.50		125
Error	± 0.332	± 0.006	± 0.004		

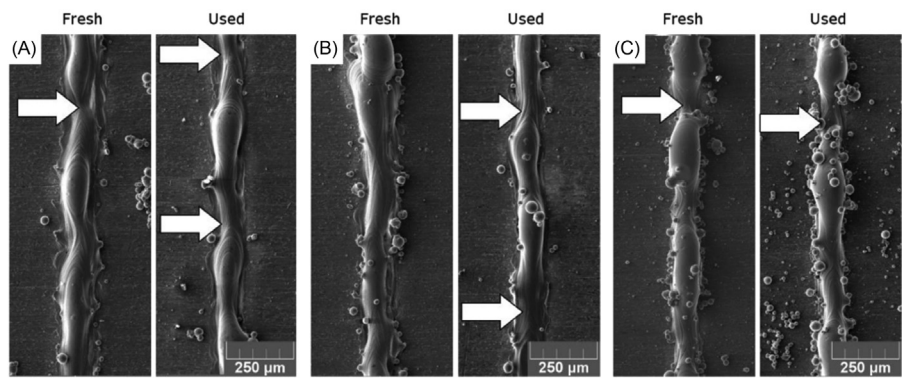


Figure 1.43 Representative scanning electron microscopy pictures of single tracks printed using “fresh” and “used” Ti6Al4V powders at a powder depth of (A) 25, (B) 60, and (C) 100 μm. *Source:* From M. Skalon, et al., Reuse of Ti6al4V powder and its impact on surface tension, melt pool behavior and mechanical properties of additively manufactured components, Materials (Basel) 14 (2021) 1–22.

Table 1.9 shows that an increase in the platform or build chamber temperature increases these effects [394]. Working in a vacuum instead of argon atmosphere (standard in E-PBF and experimental in L-PBF) leads to evaporation of aluminum [395]. Changes in chemical composition, especially the increase in oxygen, further influence the melt pool behavior. Skalon et al. [396] showed an increase in melting temperature as well as melt pool viscosity, thus leading to a decrease in melt pool stability. It impaired the instability of the singular melt tracks, as displayed in Fig. 1.43 where the melt tracks of virgin and used powder in different powder depths of (A) 25, (B) 60, and (C) 100 μm are visible. In this figure, the white arrows indicate the spots where the deposited material is missing and the cross-sections depleted. Fig. 1.44 shows an evaluation of these cross-sections, revealing a quantitative increase of the depleted sections.

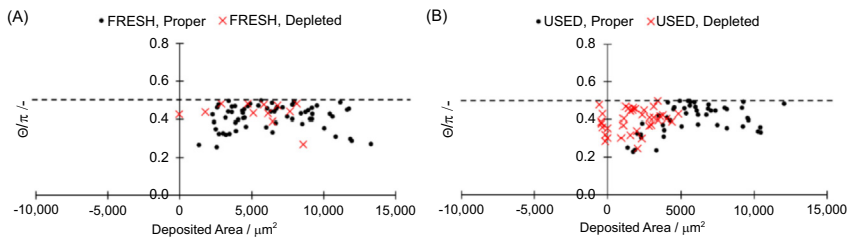


Figure 1.44 Comparison of proper and depleted melt line cross-sections shape parameters for (A) virgin and (B) used powder in the function of the area of the cross-sections.
Source: From M. Skalon, et al., Reuse of Ti6al4V powder and its impact on surface tension, melt pool behavior and mechanical properties of additively manufactured components, Materials (Basel) 14 (2021) 1–22.

Table 1.10 Relative density for parts built from virgin and used powder.

Powder	Average relative density	Relative density standard parameters
Virgin	99.5%	99.7%
Used	99.3%	99.5%

The effects on PSD, sphericity, and flowability can be transferred to materials other than Ti64 while changing the chemical composition, and its influence on melt pool behavior will strongly depend on the alloying system [397,398].

1.8.3 Influence on build parts

For build parts from Ti64, the changes in powder properties manifest in a slight decrease in overall density, as demonstrated in Table 1.10. It might be attributed to the less stable melt pool found by Skalon et al. [396]. A decrease in density is visible for nearly all sets of parameters, only a set of parameters with lower energy input (e.g., parameter set 9 and 20) are contrary to the trend (Fig. 1.45).

Since PSD and sphericity are stable, multiple reuses of powder seem to have no measurable effect on surface roughness. Fig. 1.46 shows arithmetical mean deviation *Ra* and maximum peak height *Rz* for varying parameters and surface orientation but no trend toward rougher surfaces is evitable. This might change in case of larger changes in chemical composition, which can affect evaporation and the amount of condensate in the process. Usually, the chemical composition of the parts matches that of the powder.

1.8.4 Influence on the mechanical properties

Due to changes in density as well as chemical composition, components built from used powder might vary in mechanical properties compared to their counterparts

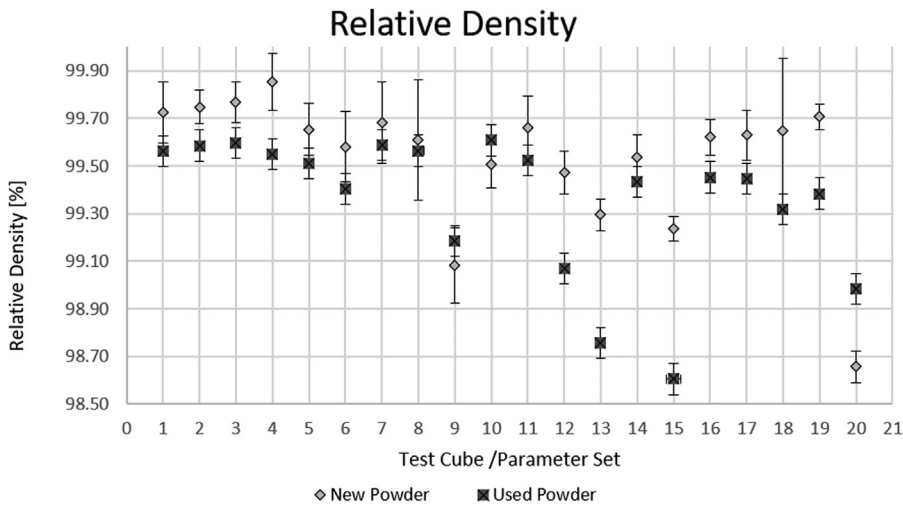


Figure 1.45 Relative density of cubes from virgin and used powder produced with varying process parameters.

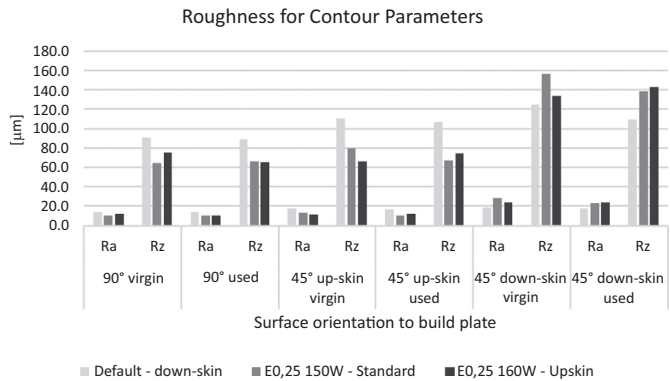


Figure 1.46 Surface roughness for virgin and used powder for varying contour parameters and surface orientation.

produced from virgin powder. Considering Ti-64 an increase in oxygen, nitrogen, and hydrogen is known to lead to embrittlement [399]. Changes in tensile strength of Ti64 reused powder are directly connected to the total oxygen content. Meier et al. [391] showed a measurable increase in strength but a reduction in elongation at break for vertically, horizontally, and specimen under a 45° to the build plate as demonstrated in Fig. 1.47. For lower oxygen levels this embrittlement effect seems not to occur as shown by Quintana et al. [392].

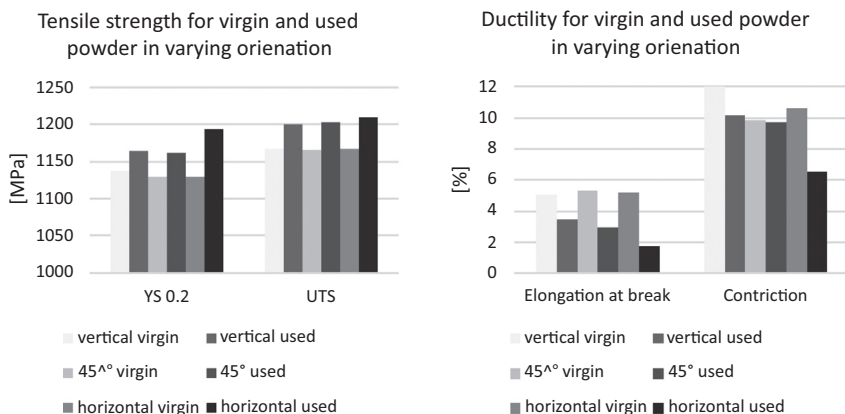


Figure 1.47 Tensile properties for virgin and used powder.

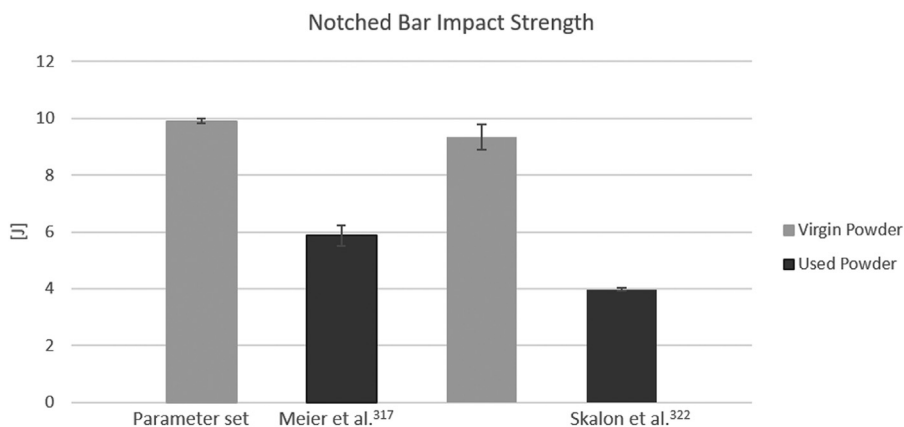


Figure 1.48 Notched bar impact test results for virgin and used powder.

Notched bar impact strength reacts more sensible to the reuse cycles and increases in oxygen, as displayed in Fig. 1.48; a decreases in impact strength from 9.9 to 5.9 J [391] and 9.35 and to 4.97 J [396], respectively, are noticeable.

1.9 Outlook: new powder-based additive manufacturing processes

1.9.1 Selective LED-based melting

An innovative approach was given regarding the energy source for powder-bed fusion 3D printing processes: LED (light-emitting diodes) instead of the well-known laser or

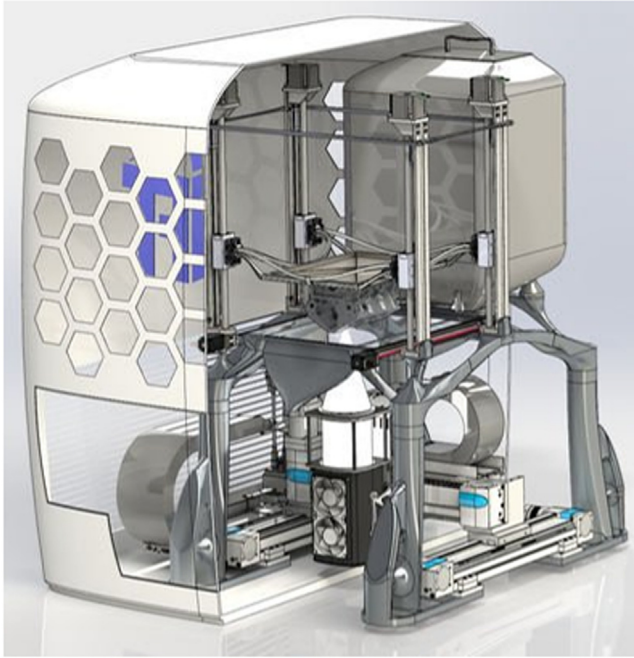


Figure 1.49 SLED sketch [400].

Source: From LED instead of laser or electron beam: new technology revolutionizes 3D metal printing at tugraz.at.

electron beam. The process, created recently at the Graz University of Technology named Selective LED-based melting (SLED)—illustrated in Fig. 1.49—is still maturing nonetheless aims to solve two core problems of powder-based manufacturing processes: the time-consuming production of large-volume metal components and the time-consuming manual postprocessing [400,401].

For achieving the energy densities required for melting metallic powders, it was designed a specially adapted LED with a complex lens system; this is necessary for adapting the focus diameter from 0.05 to 20 mm during the melting process. This capability enables SLED to melt larger volumes per unit of time without having to dispense with filigree internal structures. As an illustration, components for fuel cells or medical technology may be produced 20 times faster. Furthermore, differently from the conventional powder-bed fusion techniques, SLED adds the component from top to bottom; consequently, the required amount of powder is reduced to a minimum besides enabling necessary postprocessing during the printing process. Hence, surface smoothing and supporting structures removal are no longer necessary [400].

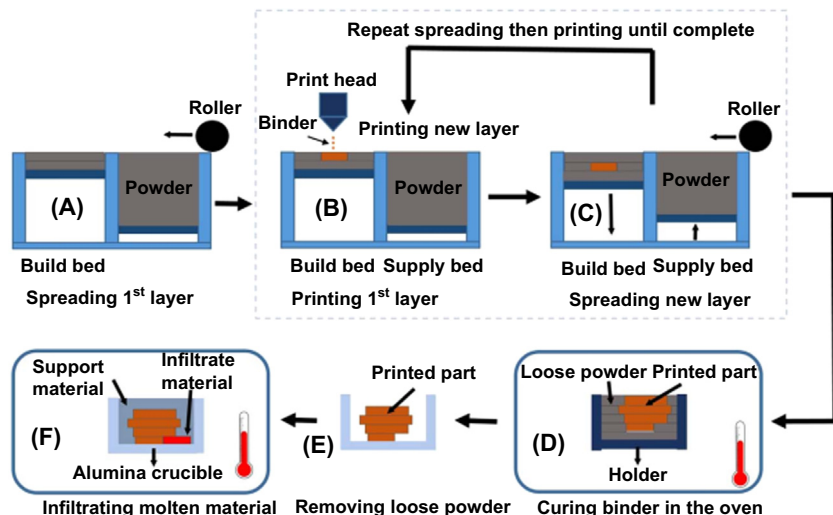


Figure 1.50 Schematics of the Binder Jetting system.

Source: From T. Do, P. Kwon, C.S. Shin, Process development toward full-density stainless steel parts with binder jetting printing, *Int. J. Mach. Tools Manuf.* 121 (2017) 50–60.

1.10 Sintering-debinding additive manufacturing

1.10.1 Binder Jetting

The process was created in 1993 at the Massachusetts Institute of Technology, being subsequently licensed 2 years later. Since then, many companies (such as VoxelJet) could start developing this technology in terms of equipment, as well as understanding the mechanisms behind the manufacturing. Fig. 1.50 illustrates the system used by the Binder Jetting (BJ) technique. In Fig. 1.50A, the roller tool supplies a powder layer for the building chamber, enabling the print head (Fig. 1.50B) to aggregate the powder and build the part layer by layer (Fig. 1.50C); a 2D pattern subsequently will turn into a 3D “green” part. Aiming to improve the strength of these parts for safe handling, a curing step; Fig. 1.50D may be required. In Fig. 1.50E, the loose powder is removed, then making it possible for the part to be densified to full density by sintering or by infiltrating external material. Due to its simplicity and nonuse of high energy density heat sources during printing, a myriad of powders (either ceramic or metallic ones) are prone to be processed by this method [402,403].

The polymeric binder is selected carefully and has to be printable. For this purpose, it must have low viscosity and be stable against large shear stresses induced by the printing. In addition, clean burnout, good powder interaction, long shelf life and acceptable environmental risk are of great interest. Focusing on the binding process itself, it can be classified as In-Liquid (the liquid that carries out the binder agent) or In-Bed (the printed liquid reacts with dry glue particles embedded in the powder bed). In-Liquid is prone to cause premature failures of print-head once the glue agent might

dry in the nozzles. Nonetheless, the agents and resins that compose the In-Liquid binder decompose thermally during postprocessing, leaving little residue. Furthermore, a wide range of powders are suitable for producing denser structures by In-Liquid binders, something not achievable by In-bed binding once voids are left in the structure after dissolving this kind of binder. In addition to the aforementioned binders, sintering inhibition binders are used for controlling the sintering by selectively jetting heat-reflective and heat-isolating materials, sintering inhibitors, and chemical oxidizers. This strategy is also employed in powder injection molding (PIM) and fused filament fabrication (FFF). However, differently from PIM and FFF, BJ has a lower binder load of $\sim 1\%$ – 10% [402,404]. After printing, the green part is subjected to curing. In this step, the powder bed and printed parts are placed in an oven at 180°C – 200°C , hence enabling the binder to be adequately dried resulting in a green part safe for handling.

Sintering is the last step, where the postcured part is heat-treated below its homologous temperature aiming to densify, strengthen it. This moment is when mass transport takes place predominantly by diffusion, reducing the surface free energy and the pore fraction in the part. Furthermore, powder features such as morphology influence the sintering behavior, whereas the shape determines the powder packing in the green state and thus how dense the sintered part will be. Also, depending on the material two ways of sintering may take place: solid or liquid state sintering [150,404]. For a better understanding of the fundamentals of these treatments, the reader is referred to check further references [405,406]. Fig. 1.51 depicts two brittle and difficult materials for processing by conventional means, and possible of being built by BJ: (A) TiAl (titanium aluminide) and (B) Ni-Mn-Ga magnetic shape memory alloy.

Regarding the process parameters and their effect on the green part, one can classify it into four groups: powder characteristics, binder, print processing parameters, and feature design as listed in Table 1.11. Each of the aforementioned effects in a different way the green and the final part, and therefore have to be

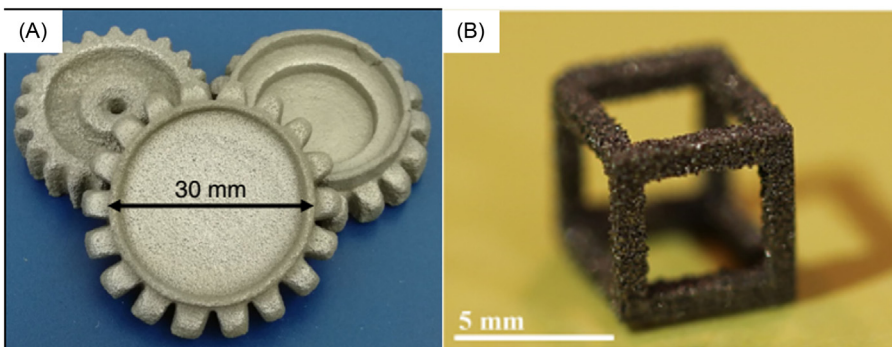


Figure 1.51 Sintered parts fabricated by BJ of (A) titanium aluminide [407] and (B) Ni-Mn-Ga [179].

Source: From M.P. Caputo, A.E. Berkowitz, A. Armstrong, P. Müllner, C.V. Solomon, 4D printing of net shape parts made from Ni-Mn-Ga magnetic shape-memory alloys, *Addit. Manuf.* 21 (2018) 579–588.

Table 1.11 List of groups and related properties affecting the green part stability.

Group	Property	Implications (e.g.,)
Powder characteristics	Shape and morphology, powder chemistry, mean size distribution, flowability, packing, and wettability	Undesirable part characteristics
Binder	Jettability and wetting behavior, viscosity and volatility of binder	Print resolution, unsafe part handling, premature failure, disintegration
Print processing parameters	Layer thickness, binder saturation, frequency of cleaning, time and temperature of curing	Inhomogeneous/weak particle interaction, reduced dimensional accuracy, residual matter
Feature design	Small pore, the thickness of support, and print resolution	Costs, postprocessing difficulties

carefully selected. A complete troubleshooting guide regarding BJ and all the possible processing implications is found in the seminal review of Mostafaei et al. [402].

1.10.2 *Metal extrusion additive manufacturing of highly filled polymers*

Metal extrusion additive manufacturing of highly filled polymers (MEAM-HF) combines metal powders and polymeric binders in filament, cartridges or pellets, allowing it to be printed on regular and relatively inexpensive additive manufacturing techniques such as the common fused filament fabrication (FFF) printers. MEAM-HF steps—printing and consolidation—are represented in Fig. 1.52. In principle the printer may be carried out by three different techniques based on the extruder and feedstock types, as follows: plunger (A. I, rod-based feedstock), filament-based (A. II), and screw (A. III, pellet). In the first case, the feedstock is delivered in cartridges being deposited layer by layer when the thermoplastic is plasticized and soft enough for extrusion. Filament-based fabrication takes place when this feedstock passes through a heated nozzle that promotes its melt whereas the extruder conducts the deposition. This method is one of the most popular in AM due to its simplicity, low cost, and safety. Since not all materials might be transformed into filament or rods, pelletization has arisen as a viable alternative. In this system, a screw carries the pellets until the melting zone aiming to soften it by heat and friction, subsequently being conducted to the metering zone in which the molten feedstock is exposed to high pressure; right after the material is moved from the feeding zone to the nozzle due to the pumping effect created by the rotating screw. One has to highlight that filler content, that is, the added powder particles to the thermoplastic binder system in these cases, are between 45% and 65%; this range makes the processing feasible and the interaction between fillers may be neglected [408]. After shaping (A), the consolidation step takes place by (B) debinding the mixture of powder plus soluble binder and backbone by solvent extraction (binder) and thermal decomposition (backbone). The last step consists

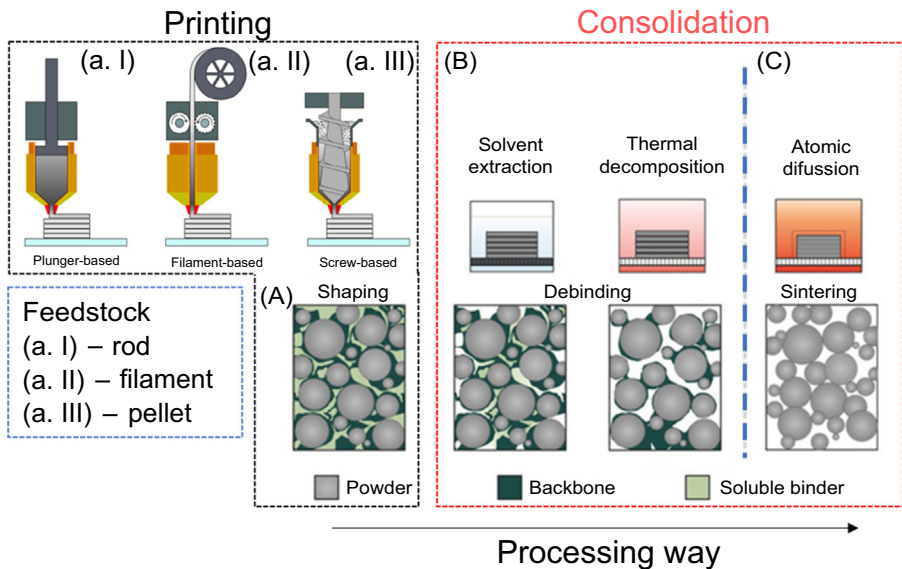


Figure 1.52 Schematics of MEAM-HF processing way: (A) shaping by (A. I) plunger, (A. II) filament, or (A. III) screw-based printing followed by (B) debinding and (C) sintering. The corresponding feedstock for each of the three techniques is indicated in the blue box.
Source: From J. Gonzalez-Gutierrez, et al., Additive manufacturing of metallic and ceramic components by the material extrusion of highly-filled polymers: a review and future perspectives, *Materials (Basel)* 11 (2018).

of the sintering, from where the part achieves mechanical resistance. Despite differences regarding deposition modes, MEAM-HF, PIM, and BJ have much in common since debinding and sintering are fundamental in these cases. Therefore, the reader is referred to check the previous section for the related sources in this topic.

Metallic materials suitable for being transformed in powder are prone to be printed by MEAM-HF, such as stainless steel [409,410], titanium alloys [411,412], tool steel [413], metallic glasses [414], and multimaterials (e.g., stainless steel combined with zirconia— ZrO_2) [415]. It shows the potential of this technique for engineering materials, as MEAM-HF enables low-cost prints when compared to other AM technologies. Therefore, room for improvement is opened and innovative ideas on feedstock production, product printing, binder system design, and equipment are required for further development [416].

1.10.3 Lithography-based metal manufacturing

Despite the name, lithography-based additive manufacturing (LBAM) differs from the well-established stereolithography. At first, instead of using a UV light for curing a photosensitive suspension consisting of dispersed metallic or ceramic particles and chemical agents, lithography-based makes use of a light source and a digital light

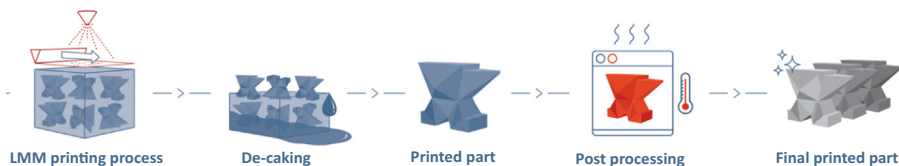


Figure 1.53 Printing, decaking, and postprocessing of lithography-based manufacturing.

Source: From incus3d.com/technology.



Figure 1.54 (A) Part printed horizontally oriented (MetShape) and (B) part with intricate design and excellent surface finishing (Incus).

Source: From incus3d.com/technology and metshape.de/lmm-technology.

processing for fabricating parts. In other words, the principle of photopolymerization is employed: the powder homogeneously dispersed in a light-sensitive resin is selectively polymerized in reason of its exposure to the light, being cured, adhering to the previous layers ensuring fixation during the complete build-up as depicted in Fig. 1.53. Further steps are related to the decaking, where the noncured photosensitive suspension is washed away aiming to leave behind the printed structure. As in the case of BJ and MFFF, LBAM requires debinding and sintering for the part to be densified resulting in satisfactory mechanical properties [417,418].

As important features LBAM does not require support structures during the printing step, thus eliminating time-consuming post processes. Moreover, the connection between the components and the building platform is absent; after decaking, the part is ready for postprocessing. Complex and small parts are capable of being fabricated with high resolution, once layers up to 40 μm are attained. Last but not least, optimum use of the printing space is possible due to the absence of support structures. It means that optimum use of this space leads to the printing of several components at the same time, whereby economic efficiency is possible [419].

Until recently most of the applications of LBAM were devoted to ceramic printing [417,418,420,421]. Nonetheless, companies such as MetShape and Incus have shown

the capability for developing lithography-based metal manufacturing (LMM) with excellent surface finishing and precision. Fig. 1.54 depicts (A) and (B) parts produced by the aforementioned companies demonstrating the feasibility of LMM.

1.11 Cold spray additive manufacturing

Cold spray (CS) is a solid-state coating technology widely applied for manufacturing, repairing, and welding parts in fields including aerospace, marine, and automotive. It has been successfully employed for a wide range of industrial

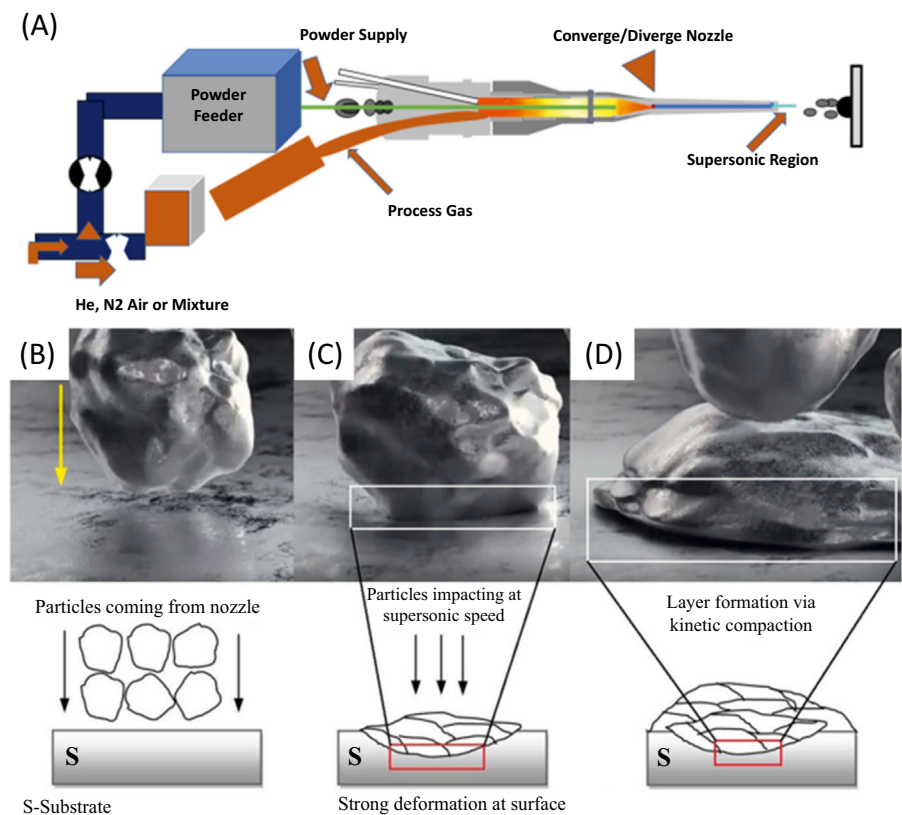


Figure 1.55 (A) schematic diagram of a cold spray gun and the mechanism behind the CSAM: in (B) the particles travel at supersonic velocity coming from the nozzle, (C) impacting with the substrate, and (D) forming the layer [427,428].
Source: From G. Prashar, H. Vasudev, A comprehensive review on sustainable cold spray additive manufacturing: state of the art, challenges and future challenges, J. Clean. Prod. 310 (2021) 127606.

materials and related alloys such as aluminum (highly reflective) [422], titanium (highly reactive) [423], shape memory alloys [424], nickel [425], and metal matrix composites (reinforced with, e.g., diamond) [426] without altering the feedstock and the underlying material properties. Only recently CS was applied as a manufacturing technique, named cold spray additive manufacturing (CSAM) illustrated in Fig. 1.55. In (A), the CS gun is depicted, while the sequent images

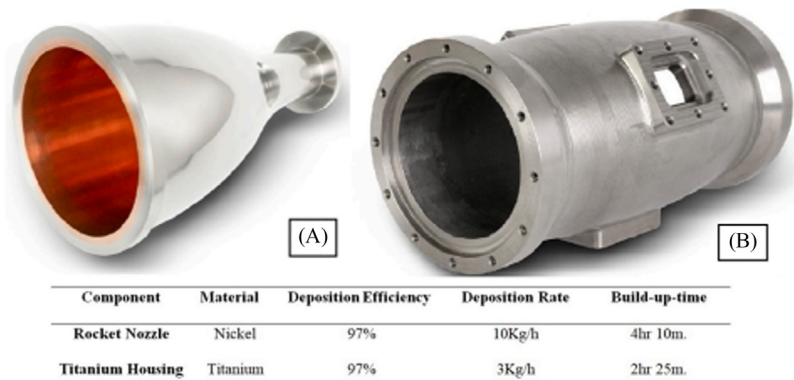


Figure 1.56 Parts manufactured by CSAM process: (A) rocket nozzle and (B) titanium housing (Impact Innovations, Germany).

Source: From G. Prashar, H. Vasudev, A comprehensive review on sustainable cold spray additive manufacturing: state of the art, challenges and future challenges, J. Clean. Prod. 310 (2021) 127606.

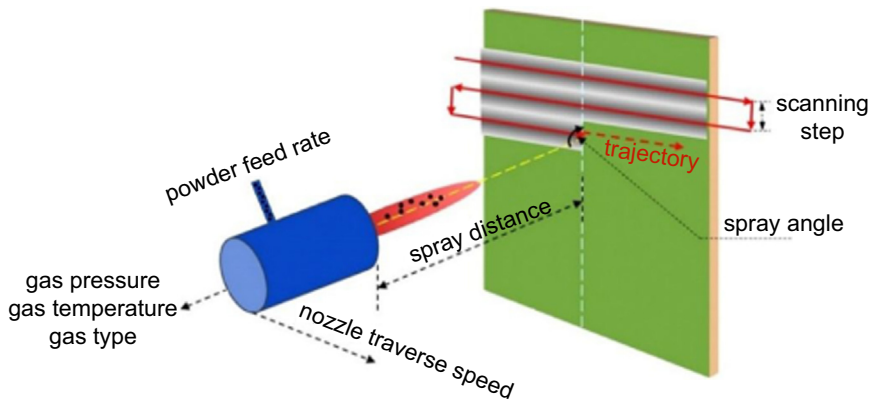


Figure 1.57 Important process parameters for CSAM.

Source: From S. Yin, et al., Cold spray additive manufacturing and repair: fundamentals and applications, Addit. Manuf. 21 (2018), 628–650.

show how CS/CSAM work. In (b), compressed gases such as air, nitrogen, or helium are used to accelerate the powder particles to velocities higher than 1000 km/h through a Laval nozzle, (B) inducing the deposition when these particles impact onto a substrate—typically a metallic one, (C) resulting in the layer formation. Differently to the conventional high-temperature processes, CSAM depositions success depends strongly on the kinetic energy rather than the thermal one before colliding with the substrate. During the collision, mechanical interlocking and local metallurgical bonding caused by localized plastic deformation between particle/particle/substrate make the deposition stable; since no melting takes place, the particles remain in solid-state and thus oxidation, phase transformation and related precipitation, and residual thermal stresses are avoided [429]. As seen, the principles behind CS and CSAM are basically the same, nonetheless while CS repairs CSAM is able to build entirely new parts.

Positive and negative aspects still make CSAM an attractive process for building 3D parts. Compared to high energy density technologies based on powder—such as electron beam melting and selective laser melting—CSAMs offer merits reparation and restoration of damaged components, low production times and higher process flexibility, and illimitable product size. Conversely, postmachining is often necessary since CSAM builds semifinished components with a rough surface, besides difficulties to control the process parameters leading to poor properties in the as-built (or manufactured) condition; the need for posttreatments for improving these properties has to be considered when CSAM is selected for printing. Last but not least, geometrical complexity is a constraint since manufacturing has been devoted to parts with rotational geometries. However, this challenge has been overcome with proper design and structural optimization [430], as demonstrated by Fig. 1.56.

According to Yin et al. [430], in general, the manufacturing parameters related to CSAM include gas pressure, temperature and type, powder feed rate, nozzle transverse speed and trajectory, spray distance, spray angle, scanning step—all indicated in Fig. 1.57. Concerning the gas variables, these influence directly the particle impact velocity and thus the properties of the deposit. It is accepted that improved deposited properties are attained by higher particle impact velocity. The powder feed rate impacts on (1) particle velocity, (2) thickness and profile of single-track deposition, and (3) residual stresses between deposits and substrate. Nozzle parameters are critical for determining the spray duration and feedstock rate (transverse speed), the uniformity of the deposit thickness and the surface morphology (scanning step), deposition efficiency (spray distance), fabrication quality represented by deposition and adhesion strength as well as cross-sectional profile (spray angle), and deposition homogeneity (trajectory). For a better overview of the effect of the aforementioned parameters on built features such as porosity, deposition strength, adhesion, residual stresses, and deposition efficiency, the reader is referred to check Table 1.12 and the work of Yin et al. [430].

Table 1.12 Process parameters and related effects on built features.

Group	Variable	If	Porosity	Deposit strength	Adhesion	Residual stress	Deposition efficiency
Gas variables	Pressure	↑	↓	↑	↑	↑	↑
	Temperature	↑	↓	↑	↑	↑	↑
Nozzle parameters	Molecular weight	↑	↑	↓	↓	↓	↓
	Powder feed rate	↑	↑	↓	↓	↑	↓
	Transverse speed	↑	↑	↑	↑	↓	↑
	Scanning step	↑	x	x	x	x	x
	Spray distance	↑	o	o	o	o	o
	Spray angle	↑	↓	↑	↑	↑	↑
	Trajectory	↑	x	x	x	x	x

↑ stands for increasing, ↓ for decreasing, “o” for relevant but no common view, and “x” no data available.

Source: Reprinted under permission of Yin et al. (2018). S. Yin, et al., Cold spray additive manufacturing and repair: fundamentals and applications, Addit. Manuf. 21 (2018) 628–650.
<https://doi.org/10.1016/j.addma.2018.04.017>.

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Directed energy deposition processes and process design by artificial intelligence

2

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2.1 Wire-arc additive manufacturing

2.1.1 Introduction

Additive manufacturing (AM) is an emerging, forward-thinking technology that offers a variety of novel and exciting possibilities alongside established and conventional processes, such as conventional joining or subtractive manufacturing. One of these AM technologies is a wire-arc additive manufacturing (WAAM), which utilizes a wire-based feedstock and an arc process to produce near-net-shape structures. Welding and fusion-based AM processes require joining metallic feedstock by significant energy input, melting, and solidification. Not surprisingly, processing by AM and welding presents similar challenges such as solidification cracking, residual stresses, and complex thermal cycling and thus microstructure formation [1].

The following section provides an overview of the possible arc techniques that can be used in WAAM, with a particular focus on Cold Metal Transfer (CMT) and gas metal arc welding (GMAW), as well as their process characteristics and benefits. Section 2.1.2 presents possibilities to modify the chemical compositions, that is, the microstructure and the mechanical properties, by different techniques and in particular by the cored wire principle. Detailed information on technical aspects, advantages and the possibility to use cored wires as a powerful tool for material development for WAAM.

2.1.2 Arc welding techniques in WAAM: cold metal transfer in comparison to gas metal arc welding

Wire arc additive manufacturing (WAAM) is a direct energy deposition (DED) technique in which an electric arc is used to melt the wire material feedstock. There

are generally three types of WAAM processes, depending on the type of heat source [2], namely (1) gas metal arc welding (GMAW)-based, (2) gas tungsten arc welding (GTAW)-based, and (3) plasma arc welding (PAW)-based.

Various techniques can be used for WAAM such as [2] GMAW [3,4], Cold Metal Transfer (CMT) [3,5], tandem [6–8], DE-GMAW [9], GTAW [10–13], and plasma [14–16]. While in plasma and GTAW processes, the filler material is introduced separately from the energy, in GMAW-based processes, the energy and material input are coupled. It means that the material input/build-up rate is directly proportional to the energy input. Volumetric AM components are usually built up over several hours and the components are subjected to intrinsic heat treatment; especially in AM, thermal management and therefore energy input play a decisive role. Thermal management is a key factor in wire arc additive manufacturing to (1) maintain an economic process, (2) mitigate heat accumulation to overcome the constraints of the deposition cycle, and (3) keep geometric accuracy and anisotropy of mechanical properties [17,18]. Especially for thermally sensitive materials with requirements on interpass temperatures/strategies (e.g., High-Strength Low-Alloy steel (HSLA) [19]) or heat treatments (e.g., hot-work tool steel [20,21]), the accurate temperature distribution, material and energy input is essential for the microstructure evolution and the final mechanical properties of the build. Generally, WAAM utilizes heat inputs of up to several hundred J/mm, and heat is typically dissipated by (1) conduction through the AM components and its substrates, (2) by convection through the shielding atmosphere or even forced convection using an additional device, and (3) by radiation to the environment [2,17,18]. There are several external techniques to actively intervene in the cooling behavior and temperature control, such as (water) cooling of the components/platform [17,18,22] or forced convection by compressed gas cooling of the AM components [23]. However, the selection of the welding power source/process is most essential for thermal management by controlling the heat input.

CMT and GMAW are both widely applied in WAAM and are characterized by their different process characteristics. GMAW is a welding process in which an arc is formed between a consumable filler wire and the metal substrate. Depending on the applied process parameters and the resulting transfer mode, average deposition rates of 3–4 kg/h can be achieved, offering high potential in the large-scale production of parts due to the high energy efficiency and deposition rates [2]. However, the GMAW is limited to a minimum wall thickness and surface finish due to a relatively large melt pool, heat input, and poor arc stability [2,24–26]. Cold metal transfer welding can be applied to overcome these constraints by reducing the burning period of the arc, and reversing the wire electrode back and forth [27–29].

CMT consists of a modified short circuit arc process developed by *Fronius* [30], being considered a new type of welding process. Even though it is included in the name, it is not actually “cold,” but merely has a lower temperature than normal GMAW processes. In the CMT standard process, the welding wire is moved in the direction of the base material (Fig. 2.1A). The digital process control detects a short circuit and the current flow is minimized by the digitally controlled power supply (Fig. 2.1B). Parallel to the interruption of the energy flow, the retraction of the

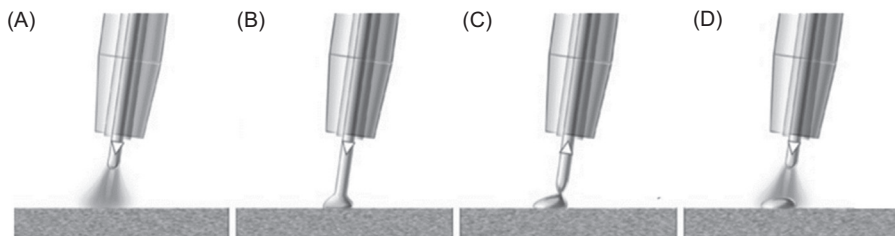


Figure 2.1 Individual phases of the standard Cold Metal Transfer process [31].

Source: From V. Wesling, A. Schram, M. Kessler, Low heat joining—manufacturing and fatigue strength of brazed, locally hardened structures, *Adv. Mater. Res.* 137 (2010) 347–374.

welding wire starts (Fig. 2.1C). In addition to the pinch effect as the main responsible factor for the material/droplet transition, the retraction of the wire and the mass inertia leads to a mechanically assisted droplet detachment at lower current levels compared to standard GMAW processes (Fig. 2.1D) [5].

During the CMT process, the arc only introduces heat for a very short time during the burn-off phase (Fig. 2.1A). The short circuit is controlled and the current is kept low. The arc length is detected and mechanically readjusted resulting in a constant length. [30] The dip transfer at a lower energy level is responsible for the spatter reduction, the lower welding temperature, and lower dilution with the substrate/preceding layers [32,33]. The lower heat input and dilution ensures a continuous deposition and prevents overheating or excessive remelting of already deposited material [33]. CMT stands out as an excellent option to reduce the energy transferred to the previously deposited layers, without reducing the deposition rate (i.e., energy input) [17].

Exceptional arc stability, drop-by-drop material deposition, controlled material transfer, low heat input, nearly spatter-free, and high process tolerance are typical CMT process characteristics, which among other benefits lead to lower reduced distortion. In general, average deposition rates of 2–3 kg/h are achievable, and the process is very suitable for the production of thin-walled components. Furthermore, challenging geometric features such as in Fig. 2.2A is also achievable, as seen in Fig. 2.2B [2]. Therefore, CMT has been selected as a promising arc-based additive manufacturing method [5,33,34] and its successful application for WAAM for various materials such as steels [25,29,35], titanium- [36,37], nickel- [38–40], magnesium- [41–43], and aluminum-based alloys [44,45] has already been demonstrated.

In WAAM, the CMT-advanced process, the enhanced variant of the original CMT standard variable polarity process, has attracted much attention recently. It combines the same controlled short-circuit technique used in the conventional CMT process, but in this case, the synergistic cycle consists of a positive electrode semicycle and a negative electrode semicycle, each consisting of a sequence of short-circuits [17]. Higher deposition rates can be realized with low heat inputs by adding an electrode negative stage [46–48]. Recent literature on the CMT-advanced process deals predominantly with aluminum alloys [17,49–52]. In addition to the

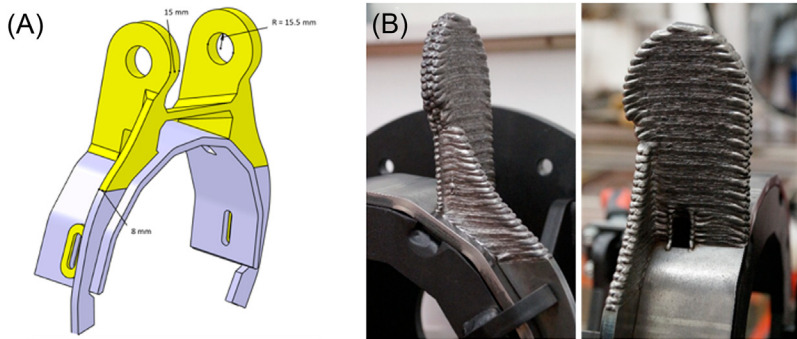


Figure 2.2 (A) CAD image of the hybrid structure for crane construction (yellow volume, manufactured by WAAM). (B) Result of manufacturing the AM part with a detailed view of the “cut-out” feature.

Source: Reproduced from J. Plangger, P. Schabhüttl, T. Vuherer, N. Enzinger, Cmt additive manufacturing of a high strength steel alloy for application in crane construction, *Metals (Basel)* 9 (2019) 1–14.

intrinsic heat accumulation issues associated with WAAM, problems related to porosity and mechanical anisotropy are reported for these materials [17]. The CMT-advanced process can reduce porosity [50] and manipulate layer dimensions in a wider range [17] compared to its conventional version, as well as additionally promotes grain refinement and introduces less heat into the substrate volume [51]. CMT in its standard and advanced form enables the build-up of AM structures with minimal energy input (i.e., dilution), increased build-up rate (Fig. 2.3), and very controlled material input (i.e., “drop-by-drop”).

2.1.3 *Materials development using filler wire: solid wire compared to metal cored wires*

In contrast to conventional joint welding, in which the filler metal and base metal are diluted in the fusion zone, the structure produced by AM consists entirely of the filler metal. Conventional filler metals are mainly designed for joint welding, not for pure additive manufacturing and thus consider dilution in the chemical composition. Due to processing challenges and the increasing market, there is a growing interest in developing new alloys specifically designed for fusion-based AM processes and especially for wire-based AM [53–55].

However, such development is technically challenging and time-consuming, which is the reason that the portfolio of additive filler material offered by welding filler metal suppliers is still limited; the filler materials mainly remain/vary within the specification for already existing grades [56]. While in powder-based processes the development/utilization of new alloy systems is considerably facilitated—since only the respective powders have to be mixed in the appropriate composition—solid wires require quite a large effort in the alloy design from melting, casting, and drawing to the final wire for

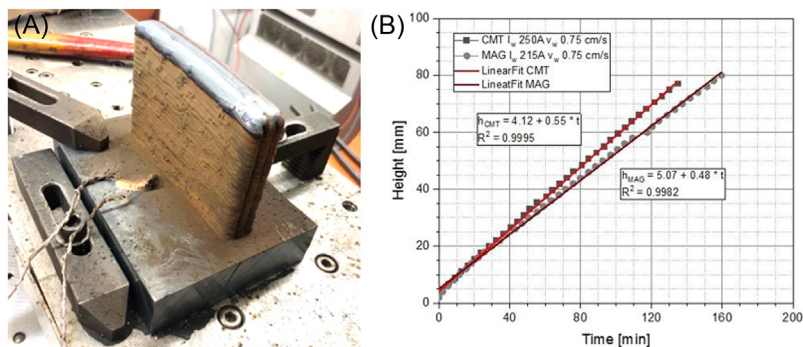


Figure 2.3 Comparison of build-up rate of CMT vs. GMAW for WAAM of hot work tool steel; interpass strategy: 350°C–400°C interpass temperature before welding the subsequent layer (A) AM test geometry with 3 welding tracks per layer and a minimum AM component height of 80 mm, (B) build-up rate for deposition of AM structure with CMT 0.55 mm/min and GMAW 0.48 mm/min at similar material deposition rate (approx. 7.4 m/min).

experimental investigation. To overcome this complex and wearing approach for wire-based processes, the development/utilization of new alloying systems uses the following approaches which have been described in the literature: (1) tandem/dual solid wire feeding [57–60], (2) particle/refiner addition to solid wire [61,62], or also (3) application of tubular metal-cored wire approach [63–66].

With tandem/double wire feeding, two filler materials are added simultaneously with varying feed rates, extending the maximum number of possible alloys. This enables a certain degree of flexibility but does not solve the actual problem, since again only standard filler material can be used (i.e., no “freedom in alloy design”). This approach is particularly suitable for (intermetallic) binary systems that are difficult and costly to produce (i.e., Ti–Al [67–69], Ni–Ti [70], Fe–Al [12]).

The addition of lower amounts of particles or refiner to the preexisting chemical composition can be incorporated directly into the solid wire [71–73], between the layer deposition by surface coating [74–77], or added into the melt pool [78]. Depending on the type of addition, the particles may be evenly or unevenly distributed. The reason/intention for the addition can be manifold: secondary strengthening, wear, nucleation sites for grain refinement, etc. Even if particles/refining additives improve the existing properties, they are an additional option and do not allow a free choice of the alloying system.

The application of cored wires is the most flexible solution in terms of alloy development and property tailoring. Research is being conducted at IMAT Graz University of Technology on the development and application of tubular metal-cored wires for WAAM [66]. While solid wires consist of a solid cross-section with homogeneous element distribution, cored wires consist of a simple standard (un-)alloyed sheath and a powder filling to adjust the chemistry (Fig. 2.4). The composition of the powder filling mixture used is determined by the target properties of the final build considering physical, chemical, and thermomechanical compatibility between the sheath and filling as an

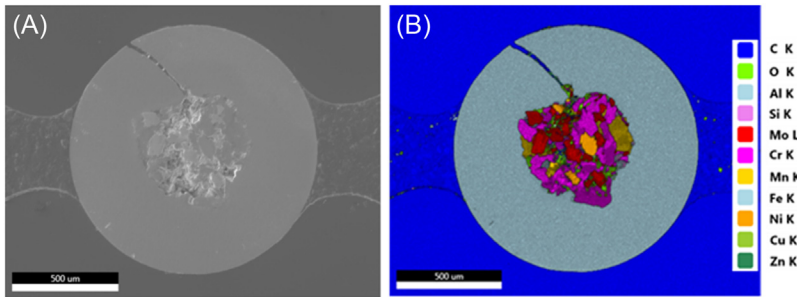


Figure 2.4 (A) Scanning electron micrograph of a fabricated metal cored wire designed for wire arc additive manufacturing and (B) associated energy-dispersive X-ray spectroscopy analysis of the powder filling and metal sheath.

essential boundary condition. In addition to metallic alloying elements, nonmetallic elements can also be introduced to improve process properties such as arc stability, viscosity, etc. as well as surface finish. The density of the powdered filler in the cored wire should not be less than 40%–50% of the theoretical density to reduce the filling of gas/air [65]. The flowability of the powder mixture in the wire should be rather low and the particle size should be varied (i.e., particle size distribution) to maximize the density of the powder filling. To prevent the opening of the cored wires under the influence of thermal exposure and mechanical stress, the sheath of the wire should be overlapped and have high stiffness. In case of low stiffness, the sheath edge can also be sealed, for example, by laser welding [65]. While solid wires are produced in larger quantities and used for serial production, cored wires can be produced in smaller batches and used for material and alloy development for AM.

To date, literature on the application of cored wires in wire arc additive manufacturing is limited [64,66]. The available information mainly deals with the usage of cored wires in classical joint welding. More commonly, metal- or flux-cored wires are processed with the spray arc during joint welding. The reduced electrically conductive sheath cross-section leads to a high current density, a deep weld penetration and, in combination with the powdery alloying elements, a high deposition rate is thus possible. Even if the maximum achievable deposition rate is a key factor in wire-based AM, near-net-shape, accuracy, or thermal management is equally important. In wire-based AM, cold metal transfer (CMT) has established itself alongside the classic metal active gas (MAG) process. This modified short-circuit arc welding process enables AM structures to be built with minimal energy input, maximum shape accuracy, and drop-by-drop controlled material transfer [29]. For the processing of cored wires using the CMT process, only a minor geometrical adjustment of the sheath cross-section (i.e., thicker cross-section for similar current density compared to solid wire) is required to facilitate short-circuiting and enable the processing of cored wires in CMT WAAM (Fig. 2.5).

Using the metal-cored wire principle, based on existing chemical compositions or completely novel compositions, new classes of materials can be developed for

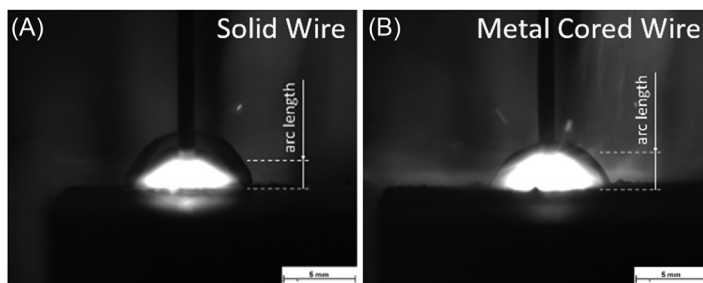


Figure 2.5 High-speed images of weld bead deposition in front view for (A) solid wire and (B) metal cored wire with cold metal transfer; similar arc characteristics are observed, with minor differences in maximum arc length and arc geometry.

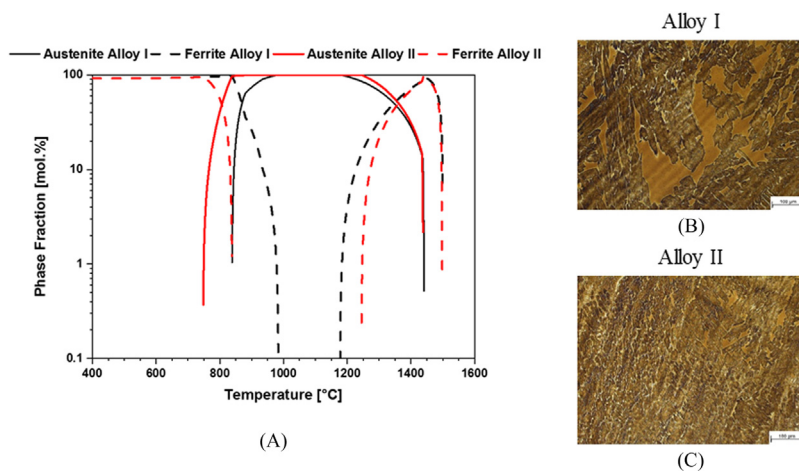


Figure 2.6 (A) Thermodynamic simulations using MatCalc for two selected hot work tool steel alloys with different nickel content (I & II) showing differences in the ferrite/austenite temperature range. (B, C) Associated microstructure of AM components fabricated with cored wires of hot work tool steel alloy I & II, where the reduction in ferrite phase fraction is noticeable for alloy II.

WAAM with CMT or MAG. The selection of the appropriate combination of alloy system for the sheath and the powder filling can be done for different materials (i.e., ferrous or nonferrous) and optimized using different simulation tools, as depicted in Fig. 2.6A. Material databases (e.g., Thermo-Calc, MatCalc, JMatPro, etc.), simulations (e.g., Scheil-Gulliver, thermodynamic, thermokinetic), or phase diagrams can be used to facilitate element selection to achieve desired properties. The addition and systematic variation of certain elements can improve the microstructural properties of the final AM components (Fig. 2.6B and C). By adding certain elements or particles,

both the microstructure (e.g., phase fraction, precipitates, etc.) and the mechanical properties (e.g., tensile strength, fatigue, notched impact strength) can be significantly influenced and optimized. The flux-cored wire concept enables complete “freedom in alloy design,” tailoring of properties and can be combined with other concepts already mentioned, such as particle/refiner addition or tandem/double wire feeding.

2.2 Wire-based electron beam additive manufacturing of titanium alloys and NiTi shape memory alloys

2.2.1 Introduction

In this section, wire-based additive manufacturing (w-EBM or EBAM) is introduced and explored focused on Ti and shape memory alloys (NiTi). At first, an overview of the technique is given, where general statements regarding the operational mode, processing parameters and its implications on the produced parts plus the challenges related to the selection of the proper ones, and material transfer mode are briefly addressed.

2.2.2 Wire-based electron beam additive manufacturing

Directed Energy Deposition (DED) additive manufacturing (AM) techniques comprise all processes where focused energy generates a melt pool into which feedstock is delivered by a nozzle. As heat source laser, electron beam or arc is employed, whereas feedstock powder or wire is suitable. According to Milewski [79], wire-based DED processes using an electron beam as a power source may be referred to as Electron Beam Freeform Fabrication (EBF3) or wire-based Electron Beam Additive Manufacturing—w-EBAM [80], used from now on. As main purposes, these technologies may be used to support fabrication and repair of large space structures, spacecraft primary structure, and replacement components. Fig. 2.7 depicts the schematic of w-EBAM primary components and operation mode. In a nutshell, the process works by introducing a melted wire into the molten pool, which is created and sustained by a focused electron-ion beam in a high vacuum chamber. Gradually this substrate is translated for both electron beam and wire and, on each step, a layer is deposited resulting in the built structure.

In reason of their versatility, these cutting-edge technologies have gained importance in DED processes, achieving increasingly more acceptance for applications [82,83]. Furthermore, wire-based DED processes [e.g., wire arc additive manufacturing (WAAM), electron beam additive manufacturing] are broadening the scope of application and have received considerable attention in the industry due to the printing of more voluminous structures while maintaining high deposition rates. w-EBAM can be classified as a near-net-shape manufacturing process with the highest deposition rates up to $2500 \text{ cm}^3/\text{h}$ and usually requires additional finishing with some kind of machining [84]. Since the objective is to achieve the lowest

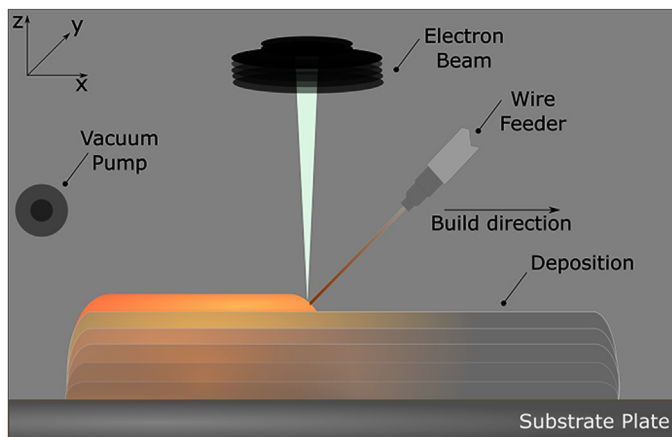


Figure 2.7 Schematics of the w-EBAM process [81].

Source: From R. Guimarães, et al., The electron beam freeform fabrication of NiTi shape memory alloys. Part I: Microstructure and physical–chemical behavior. *Proc. Inst. Mech. Eng. Part. L J. Mater. Des. Appl.*, 146442072097505 (2020). <https://doi.org/10.1177/1464420720975059>.

possible dilution, the high power density of up to 10^7 W/cm^2 characteristics of EB is not required, as deep penetration and a keyhole effect are not necessary [85,86]. At present, w-EBAM has found its application to produce AM parts from steels [29,87,88], titanium-, aluminum- [1,44,89], or nickel-based alloys [90–92] at industrial scale on commercial systems [93,94] and the potential use to repair components [95].

However, titanium and its alloys are of particular interest to the aerospace industry due to their unique properties such as high strength-to-density ratio [96]. The production route of such alloys is very demanding, costly, and associated with significant challenges. To overcome these challenges, a high material-utilization ratio represented, for example, by the “buy-to-fly ratio” should be aimed for, which can be achieved by utilizing AM processes [97]. The number of suitable AM processes is limited and the requirements for the shielding environment are remarkably high to avoid atmospheric contamination by interstitial elements such as oxygen. Dissolved oxygen in the titanium matrix leads to an increased strength by solid solution hardening but also an undesired significant reduction in fracture elongation [98,99]. Electron beam processes seem to be more suited since the process takes place under vacuum ($<5 \times 10^{-3}$ mbar) and contamination by oxygen can be minimized [100]. The suitability for processing titanium and its alloys by w-EBAM has already been reported in the literature [86,95,101–104].

In w-EBAM, numerous process parameters such as (1) accelerating voltage, (2) beam current, (3) welding speed, (4) wire feed speed, (5) beam figure, and (6) focus position can be independently varied over a wide range to control the process; proper selection of process parameters remain a challenge [86,105]. Since the melt pool and its temperature distribution can be controlled by process parameters

[106–108] and weld beads form during solidification, knowledge of the relationship between process parameters and weld bead geometry is essential for process control [86]. Pixner et al. [86] correlated the process parameters with the resulting weld bead geometry. While the beam current and beam figure diameter significantly influence the weld bead width, the material input per unit length (i.e., the ratio of wire feed rate to welding speed) determines the weld bead height. Dilution can be minimized by reducing the beam current, but still providing sufficient energy input to melt the fed wire material and some substrate/previous layer. Based on the weld bead geometry and the knowledge of the individual weld bead profiles, the possibility to find an optimal overlap distance and welding sequence arise. These are important specifications for the further building process and the design/manufacturing of more complex geometries [86,109].

A further consideration in process design is the type and direction of wire/material feeding. Unlike WAAM, where the material input is usually coupled with the heat source (arc), in w-EBAM, the material input and the energy input are decoupled. Therefore, the parameters for material (e.g., wire feed rate) and energy input (e.g., beam current, accelerating voltage, welding speed) can be selected separately, but can only be varied independently of each other to a limited extent, since a certain energy input per deposited material volume is required [86]. The correct positioning/height alignment of the wire tip with the electron beam heat source is mandatory for material transfer and a stable weld pool [110]. Fig. 2.8 illustrates two modes of material transfer: an avoidable (droplet transfer, Fig. 2.8A) and a preferable (liquid metal bridge, Fig. 2.8B) one. When the spacing between the wire tip and the molten pool becomes too large, the liquid metal bridge changes to a droplet transfer mode. Correct positioning results in a continuous flow of metal from the molten wire to the melt pool by a “liquid metal bridge” [111,112]. While the preferred liquid metal transfer mode results in a smooth surface finish, droplet transfer results in increased spatter and rough surface.

The mechanical properties of AM components are mainly influenced by three factors and are as follows: (1) element segregation, (2) volume defects, and (3) microstructure [113,114]. The volume defects are primarily intrinsic porosity [80,115,116], cracking due to residual stresses generated during thermal cycling of

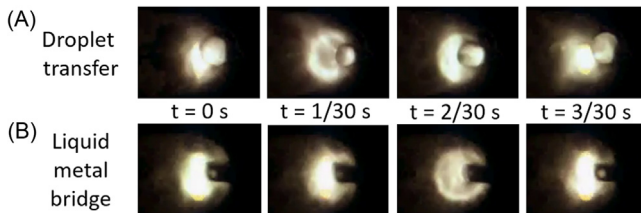


Figure 2.8 Material transfer modes in wire-based electron beam additive manufacturing recorded via CCT camera (A) droplet transfer and (B) liquid metal bridge transfer.

Source: From F. Pixner, et al., Wire-based additive manufacturing of Ti-6Al-4V using electron beam technique, *Materials (Basel)* 13 (2020) 3310.

the process, and delamination due to insufficient melting [116]. In particular, insufficient bonding (lack of fusion) as a predominant defect between the weld tracks adversely reduces the static and dynamic mechanical properties [117,118].

2.2.3 Wire-based electron beam additive manufacturing of titanium alloys

All AM processes have complex, multiple thermal cycles and transient temperature profiles in common, as depicted in Fig. 2.9 for the first (layer 1) and the fourth (layer 4) layer of a Ti-6Al deposition; here, a difference in thermal profile—thus thermal gradients—is noticed by the length of the thermal zone (red), thus exposing the thermal gradient inherent to layering. In AM processes, the microstructure (e.g., including prior β -grains, martensite, and α -phase morphology for $\alpha + \beta$ -alloy) depends on the experienced thermal cycle induced by the process. The variety and diversity of microstructure have a significant role in the mechanical properties of titanium alloy AM parts [96,119].

Ti-6Al-4V is recognized as the most popular $\alpha + \beta$ -titanium alloy, covers nearly half of the titanium market share, and is the main focus in AM of titanium alloys [120]. It is (commercially) processed by different AM techniques such as powder-based processes, laser powder bed fusion (L-PBF), electron beam powder bed fusion (E-PBF), or wire-based DED techniques. Each of the respective processes has specific characteristics and thermal cycles, which result in intrinsic properties of the AM part produced. In L-PBF, cooling rates of more than 1000 K/s [80,96,121,122] can be achieved during layering, resulting in a predominantly martensitic microstructure for the as-built parts. Post heat treatment can decompose the microstructure into $\alpha + \beta$ [80], resulting in a fine lamellar, colony, or Widmanstätten- α structure and a small amount of β -phase [113]. For E-PBF, the required prevailing vacuum (i.e., absence of convection) during layer-by-layer build-up can promote heat accumulation and lower cooling rates than for L-PBF [123]. The microstructure can be already decomposed by the thermal cycling during the build-up process in E-PBF [123–126].

In general, the macrostructure of AM parts manufactured by wire-based DED processes (Fig. 2.10A) consists of epitaxially grown, columnar, prior β -grains extending over several layers and oriented in the build direction [001] (Fig. 2.10B). The

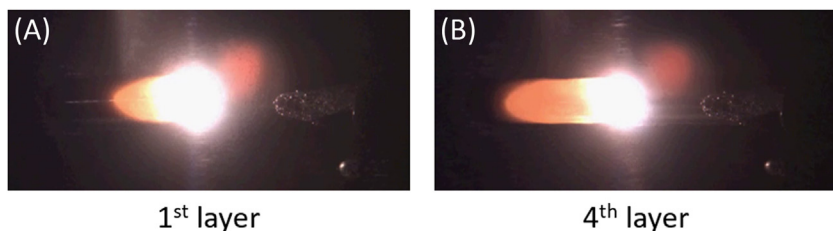


Figure 2.9 Transient temperature profiles in wire-based electron beam additive manufacturing recorded via CCT camera, depicted by single-track multilayer AM wall: (A) 1st layer and (B) 4th layer.

characteristic shape and size of the columnar prior β -grains is caused by the large temperature gradient during solidification and following the largest temperature gradient [5,86,127]. The size of the prior β -grains can be influenced to some extent by process parameters, but also by the addition of particles responsible for the nucleation of multiple β -grains, resulting in refinement [74]. Layer bands (dark/white etching contrast in Fig. 2.10A) characterize the transition from one weld bead to the other and represent minimal microstructural changes (HAZ) due to thermal cycling and self-heating of the adjacent weld bead deposit during the actual build-up process (Fig. 2.10C and D) [5,86,128–130]. Microsegregation occurs near these layer bands and are mainly related to β -stabilizer elements Fe and V [130].

For processes with the highest deposition rates and high energy input (i.e., DED processes), cooling rates tend to be low and $\alpha + \beta$ can form directly into basket-weave, Widmanstätten, or lamellar α -morphologies [80,128,131–135]. For w-EBAM, there is limited information on a systematic investigation of the influence of process parameters (e.g., energy per unit length) and build-up strategy (e.g., continuous or discontinuous) on the microstructure. Microstructural evolution and mechanical properties vary depending on the applied process parameters and build-up strategy [86,95,102,104,136–139]. For example, while Xu et al. [102] showed that at

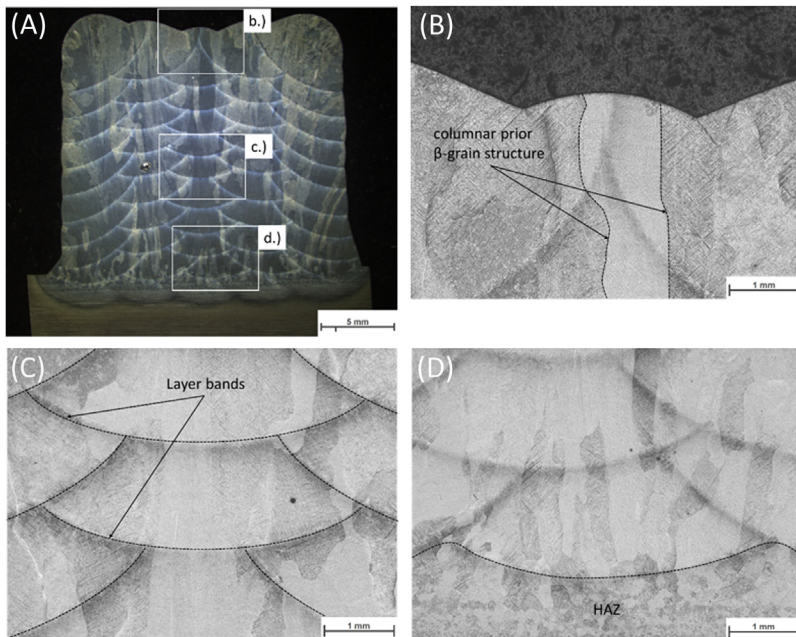


Figure 2.10 (A) Macrostructure with layer bands and columnar structure, (B) columnar prior β -grain structure, (C) presence of several layer bands, and (D) transition AM bulk material to heat-affected zone (HAZ).

Source: From F. Pixner, et al., Wire-based additive manufacturing of Ti-6Al-4V using electron beam technique, Materials (Basel) 13 (2020) 3310.

comparably high energy input per unit length and continuous build-up, the fabricated Ti-6Al-4V samples consisted of coarse prior β -grains and a graded lamellar/basket-weave/Widmanstätten structure, Pixner et al. [86,104] observed a mixture of martensitic and finer α -lamellar structure within smaller prior β -grains (Fig. 2.11A) at comparably low energy input per unit length, discontinuous build-up, and cooling rates of 180 to 350°C/s in the temperature range of the $\alpha + \beta$ field. Klimenov et al. [136] varied the build-up sequence [deposition algorithm: (1) discontinuous linear deposition and (2) continuous zigzag deposition with 90° rotation each layer] to show the influence on the microstructure formation. While in the later continuous deposition algorithm, the prior β -grains contained the basket-weave Widmanstätten microstructure, in the discontinuous linear layer-by-layer deposition algorithm, the microstructure consisted of α -, α' - and residual β -phases with different volume ratios. Consequently, not only the microstructural features but also the mechanical properties such as yield strength, tensile strength, and hardness changes [136].

w-EBAM has a characteristic attribute; since the process utilizes a concentrated energy source and the process is conducted under vacuum conditions, selective vaporization and loss of alloying elements may occur depending on its vapor

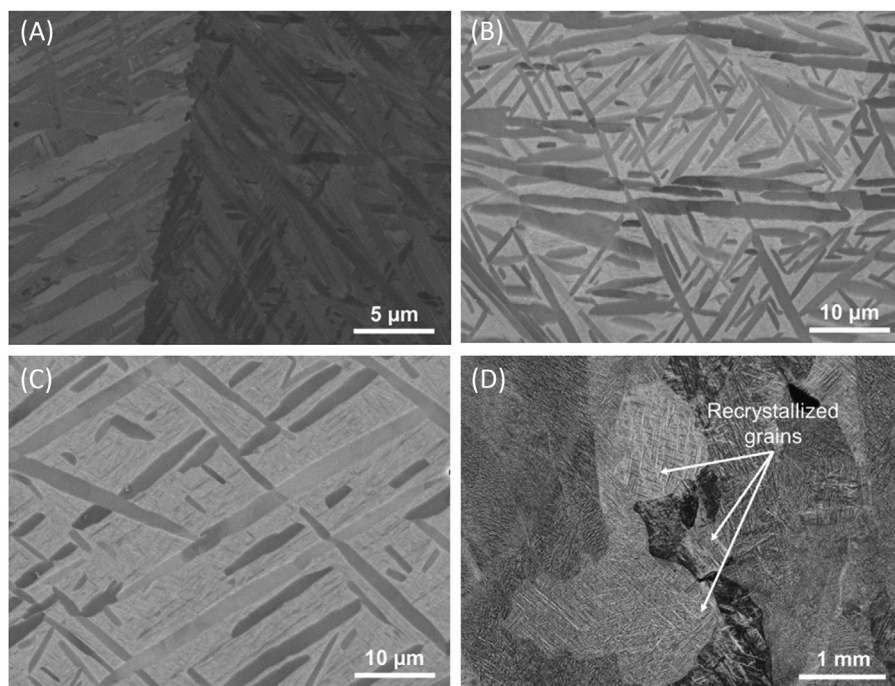


Figure 2.11 Example of microstructure in AM components of Ti-6Al-4V: (A) As-deposited condition, (B) postheat treatment at 870°C, 60 min, and water quenched, (C) postheat treatment at 910°C, 60 min, and water quenched, and (D) partial recrystallized grains after post heat treatment at 1010°C, 10 min, and water quenched.

pressure. Aluminum is the element in Ti-6Al-4V with the highest saturated vapor pressure, it is expected to have a significant tendency to vaporize. The evaporation loss of aluminum during the processing of Ti-6Al-4V by different AM processes has been reported in the literature, for example, EBF3 [102,140], E-PBF [141,142], or L-PBF [143]. However, excessive evaporation losses occur mainly in vacuum-based processes (E-PBF up to 30% [142] and EBF3 up to 39% [102]). Pixner et al. [86] showed that by minimizing dilution, that is, avoiding excessive remelting of previous layers, excessive evaporation of aluminum (reduction to approx. 14%) can be prevented. Nevertheless, it has to be considered that the chemical composition of the AM parts changes noticeably compared to the filler material.

In general, AM DED processes focus on Ti-6Al-4V ($\alpha + \beta$ alloy), which has been processed with w-EBAM so far [144]. The literature on the other groups of titanium alloys is still scarce. Recently, Zhang et al. [144] investigated the microstructural evolution and associated properties of the near- β alloy Ti-17 (Ti-5Al-2Sn-2Zr-4Mo-4Cr) in different conditions. The microstructure of the as-deposited alloy contains also coarse columnar prior β -grains epitaxially growing from the substrate. The microstructure within the coarser grains consists of intragranular ultra-fine acicular α_p , continuous α_{GB} , and retained β .

The use of different post heat treatments in as-deposited titanium alloys can help to tailor the mechanical properties of the component. Solution treatments at subtransus temperature are used usually for homogenization of the microstructure and relief of the remnant residual stresses due to the cooling. The temperature and time in this region and the cooling rate vary the width of the α lamellar and the stabilization of β phase, affecting the tensile strength of the material [145]. Fig. 2.11B and C shows an example of a change of the width of the α lamellar after post heat treatment in Ti-6Al-4V at 870°C and 910°C. The combination of the solution treatment with ageing in Ti-6Al-4V may assist in the precipitation of Ti_3Al , which reduces the ductility of the material. Solution treatments in the β -region can produce partial or fully static recrystallization followed by grain growth, depending on the process parameters and the temperature of the heat treatment (Fig. 2.11D). In the case of near- β titanium alloys, post heat treatments below the beta transus temperature would help to control the morphology, the volume of α phase, and the precipitation of secondary α phase to meet microstructure-mechanical properties relationship similar to wrought materials [144].

2.2.4 Wire-based electron beam additive manufacturing of NiTi shape memory alloys

Shape memory alloys (SMA) are intermetallic alloys with shape memory effect (SME) and superelasticity (SE), both resulting from a reversible solid-state transformation. Overall, this class of materials are known for its ability to recover its previous shape after being deformed (1) when heated, prompting the occurrence of phase transformation from martensite to austenite (or the SME), or (2) when the stress is released in the austenitic phase field, reverting the stress-induced martensitic

transformation (SE). The uniqueness of these transformations comes from the large recoverable strains (up to 12%) resulting from them, raising also significant stresses when the material is sufficiently constrained. The strain generated by these transformations (or transformation strain) can be added to the ones generated by the standard thermoelastic behavior, leading to high actuation forces and displacements. For this reason, SMAs are included in the class of smart materials [146]. Among the known SMAs, NiTi is the most investigated in reason of its light weightness, density, biocompatibility, and superior mechanical properties (thus actuation forces).

The difficulties found in the machining and forging of NiTi made AM an ideal alternative. Moreover, given the degree of freedom provided by the AM techniques, NiTi parts have expanded their niche of applications being considered for applications other than biomedical ones. Recent reviews have shown that powder-based techniques—such as selective laser melting and laser-engineering net shape—are the most employed, reaching excellent results regarding mechanical properties and microstructural stability [147,148]. However, despite the success, some limitations concerning part size and deposition rate remain open; it gives the possibility for wire-based techniques to be explored as an alternative.

Currently, investigations on the manufacturing of NiTi by WAAM have been well explored (e.g., [149–151]). Wang et al. [149] disclosed the first work on this topic printing NiTi using a dual wire feeder with pure Ni and Ti, aiming to achieve a Ni-rich Ni_{53.5}Ti (at.%) composition. A significant influence of the thermal history during the build-up was reported to affect the phase transformation significantly, leading to an anisotropic microstructure composed of Ni₄Ti₃ (bottom) and Ni₃Ti (upper part). As a consequence, it was noticed a decrease in the transformation temperature (martensitic starting temperature, M_s) along the build direction; ductility followed the same trend, differently from hardness and tensile strength. Differently from the aforementioned authors, Zeng et al. [150] produced NiTi parts using a Ni-rich NiTi wire. The built parts presented superelastic behavior at room temperature under tensile conditions, exhibiting a stable mechanical response after 7 load/unload cycles. Despite remarkable chemical differences on the layers thus a distinct functional behavior along the build direction, these works express how WAAM has increasingly proved its feasibility for additively manufacturing NiTi.

As in the case of WAAM, w-EBAM of SMA have been only recently reported in the literature [81,152,153]. The purpose of the first published work was to compare the superelastic performance of parts produced by w-EBAM and laser-engineered net shape (LENS) [152]. Regarding w-EBAM, a Ni-rich Ni₅₁Ti wire was employed as a feedstock, whose processing parameters are as follows: accelerating voltage of 60 kV, beam current of 15 mA, feeding speed of 100 mm/min, and welding speed of 2000 mm/min.

Fig. 2.12A shows the differential scanning calorimetry (DSC) for both as-deposited and heat-treated (500°C for 2 h) samples, followed by the superelastic behavior assessed in compressive mode at room temperature for as-deposited and aged, and at 53°C (austenitic finishing temperature (A_f) + 20). When in as-built state, the chemical inhomogeneity takes place together with residual internal/thermal stresses, both inherited from the high-energy-density process/solidification

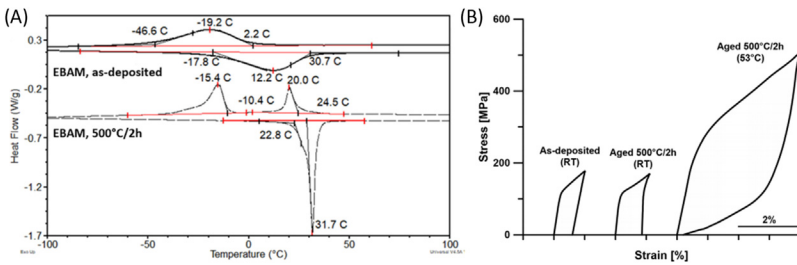


Figure 2.12 (A) Differential scanning calorimetry of as-deposited and aged (500°C, 2 h) w-EBAM samples, and (B) compressive assessment of the superelastic behavior of as-deposited, and aged at room temperature and $A_f + 20^\circ\text{C}$.

Source: Modified and reprinted with permission from J. Dutkiewicz, Ł. Rogal, D. Kalita, M. We, Superelastic effect in NiTi alloys manufactured using electron beam and focused laser rapid manufacturing methods, *J. Mater. Eng. Perform.* (2020). <https://doi.org/10.1007/s11665-020-04938-z>.

conditions; also, grain size plays an important role. Therefore, a shallow DSC peak is seen meaning a gradual phase transformation. On the other hand, aged samples presented sharp peaks resulting from an energetically favorable transformation due to the residual stresses recovery and R-phase precipitation processes. The mechanical behavior demonstrated in Fig. 2.13B for both as-deposited and aged samples at room temperature have negligible superelastic effect since the plastic deformation remains after the stress is released. However, when tested in the austenitic phase field, the aged sample behaves accordingly, exposing an almost complete strain recovery up to 4% after one cycle [152]. All in all, the authors concluded that either w-EBAM or LENS are suitable for fabricating functional NiTi parts.

Guimaraes et al. [81] conducted a study to evaluate the influence of w-EBAM on the build-up of single-wall construction with 1, 5, and 10 layers. For this case, it was employed the following parameters: 90 kV of accelerating voltage, 22.5 mA of beam current, 10.5 mm/s of feeding speed, and 2.85 m/min of feeding speed. The height increased linearly, while the width presented an asymptotic behavior following the findings of Pixner et al. [86] for Ti-64. Moreover, energy-dispersive X-ray analysis revealed slight variations in Ni composition along the built direction, achieving a relative stabilization in the last layers. Besides, the microstructure was cellular in reason of the thermal history, presenting Ti-rich eutectic and Ti_2Ni precipitates. The DSC analysis revealed favorable transformation in the as-built condition, meaning that the energy employed during the process was suitable for fabricating functional samples. The same procedure was used to determine the M_s for carrying out the mechanical assessment of the superelasticity. At $A_f + 15^\circ\text{C}$, four conditions were cycled 10 times at a constant strain rate of $10^{-4}/\text{s}$ and unloaded at 100 N/s in compression mode. Fig. 2.13 depicts the results for (A) as-built, (B) solubilized at 950°C for 6 h, (C) solubilized and aged at 350°C at 1 h, and (D) solubilized and aged at 450°C for 1 h. One can notice the distinct behavior resulting from the solubilization treatment if compared to the as-built sample in the first cycle: while the former recovered 60%

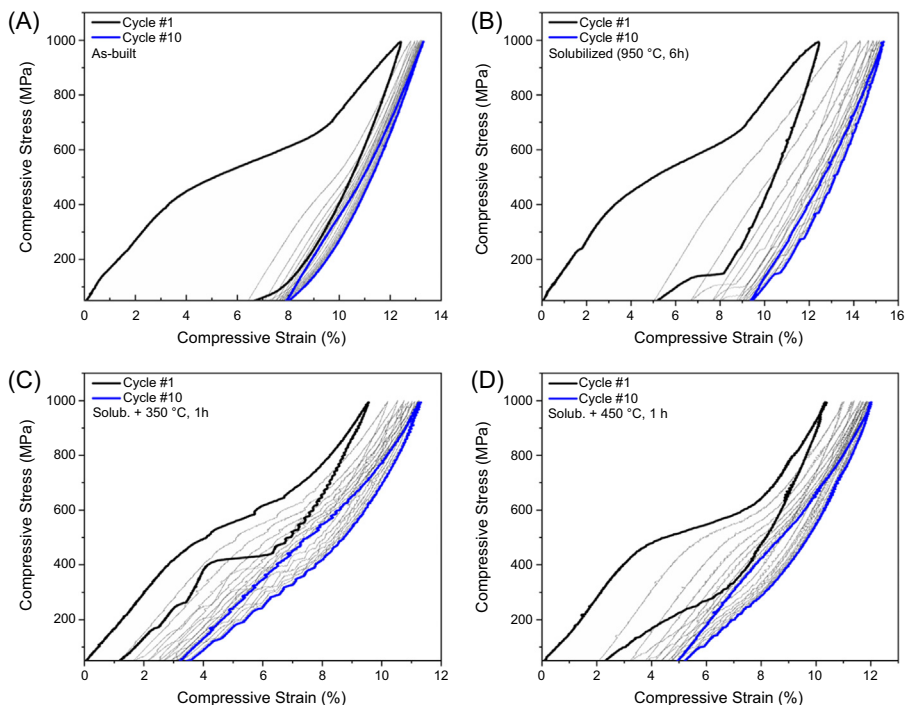


Figure 2.13 Superelastic behavior evaluated in compressive mode for (A) as-built, (B) solubilized at 950°C for 6 h, (C) solubilized and aged at 350°C for 1 h, and (D) solubilized and aged at 450°C for 1 h. Tests conducted at (A) 35°C, (B) 31°C, (C) 62°C, and (D) 70°C. Strain rate of 10^{-4} /s and unloading rate of 100 N/s.

the latter achieved 48%. However, after 10 cycles both presented similar strain recoveries: $\sim 40\%$. The ageing contributed to the precipitation of coherent Ni_4Ti_3 particles that works as a nucleation site for the stress-induced martensite inherent to the superelastic behavior. Ageing at different temperatures and at the same time allows for evaluation of the effect of the particle growth, which led to differences in the strain recovery. One can notice that the ageing at 350°C promoted 88.6% of strain recovery after the first cycle (vs. 79.8% of 450°C), stabilizing with 71.2% (vs. 57.5%) after the tenth cycle; a permanent strain of 3.5% was found (vs. 5.3%). Therefore, in consonance with the results presented by Dutkiewicz et al. [152] regarding superelasticity after ageing w-EBAM NiTi samples, this property shows itself satisfactory posttreatment. Nonetheless, w-EBAM needs room for improvement aiming to produce functional parts ready for employing without posttreatment (as already achieved by selective laser melting [154]).

2.3 Outlook: new wire-based additive manufacturing processes

2.3.1 *Resistance welding additive manufacturing (or Joule Printing)*

Resistance-based additive manufacturing has opened an important door with Joule Printing, a fast and low-cost process based on the Joule heating (or resistance heating) created by Digital Alloys. The process, developed recently, was conceptualized aiming to reach the following benefits: (1) low production costs since the process employ inexpensive raw material and high printing speeds, and (2) repeatable quality [155,156]. It works based on the rapid and precise movement of the wire feed system, positioning the tip of the wire in contact with the desired location (or melt pool). When the position is reached in the printing bed, the system pushes current through the wire making it possible to melt it by resistance heating. By moving the printing head and keeping the continuous material flow/fusing the layers, a fully dense metal part is achieved.

Additionally, the melt pool size (and consequently the dilution) plays an important role for AM. Despite not specified the processing conditions for the former (i.e., if the process is optimized or not), the latter seems to show shallower melt pools and thus lowed dilution values—one has to consider that Joule Printing uses 0.9 mm diameter wires, whereas other wire-based DED processes typically use 1–3 mm. It means that more material is employed for building the sample, and the material wasting is decreased. Furthermore, the deposition with Joule Printing allows a better resolution if compared to wire-based DED, according to the manufacturer.

According to Digital Alloys (Joule Printing, 2019), positioning and melting of the wire in a single step, simultaneously, reduces radically the printing time, assure the repeatability and therefore impairing on the costs. In other words, using an effective way for converting electrical energy into heat, a feedstock that does not require handling or special environment, and an accurate positioning of the print head makes possible a trustable and inexpensive 3D printing process. Using inexpensive commodity wires, a deposition rate of 5–10 kg/h using a very low power (<1 kWh/kg) is feasible (Joule Printing, 2019). Despite still under development, this additive manufacturing method shows itself simple, economic, and attractive.

2.3.2 *Liquid metal additive manufacturing*

This manufacturing technology, based on liquid metal droplet formation, started to be developed at the beginning of the 2000s by Canon Océ. Under the name of “Device of ejecting droplets of a fluid having high temperature” [157], a patent was required considering metals with melting points up to 2000°C. Here, a piece of equipment consisting of a chamber, where the metallic feedstock is melted, is surrounded by a magnetic coil that applies a magnetic field into this liquid. Considering an electrically conductive liquid, the magnetic field induces an

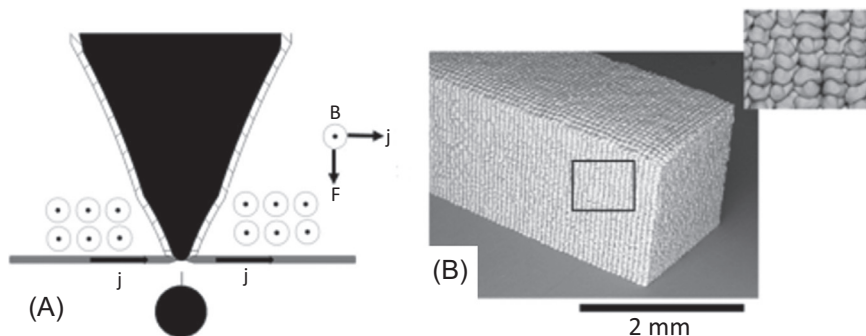


Figure 2.14 (A) The MetalJet ejection mechanism and (B) printed Ag part.

Source: Modified from M. Simonelli, et al., Towards digital metal additive manufacturing via high-temperature drop-on-demand jetting, *Addit. Manuf.* 30 (2019) 100930.

electrical current into it creating a Lorentz force. This force acts in driving the molten metal into the print's head orifice, subsequently ejecting the drop, as seen in Fig. 2.14A; a resulting Ag printed structure is seen in (B) [159]. After further developments, a partnership led to the development of the drop-on-demand metal jetting technique named MetalJet, capable of depositing microdroplets ($<80\text{ }\mu\text{m}$) of high-temperature metals ($>1000^\circ\text{C}$ melting point) to build-up near net shape structures without the need of postprocessing machining [158].

A different configuration of the same technology was patented in 2017 under the name of "Conductive Liquid Three Dimensional Printer" [160]. In this case, a radially inward Lorentz is applied on the molten metal in reason the DC pulse resulting created by the coil. A droplet is expelled from an orifice in conjunction with pressurized inert gas, achieving drop-on-demand printing rates up to 1000 Hz. Since 2019 Xerox Corp. owns the rights of the process, depositing several Al alloys successfully.

2.4 Friction-based additive manufacturing

One of the most important questions concerning the AM of metals resides in the side effects brought by the high process temperatures on the 3D-printed part. As already discussed, the vast majority of AM processes resort to temperatures that must necessarily surpass the melting point of the feedstock material (either powder or wire), since those processes rely fundamentally on manipulating the melt pool characteristics to manufacture a given model. However, melting and solidification cycles tend to result in undesirable microstructural features, which included, but are not limited to: porosity due to either entrapped gas or incomplete fusion, crack formation due to the steep gradients present upon cooling, nonmetallic inclusions due to melting pool oxidation, microstructural segregation and others [161–165].

Recently, the use of frictional heat to produce metal parts layer-by-layer has been explored as an alternative to circumvent the aforementioned issues. In general

terms, this strategy would rely on the heat generated by rubbing two metallic bodies in direct physical contact with each other, which causes their softening and allows them to be shaped by external forces [166]. Such an idea does not require surpassing the melting temperature of either rubbing body to function properly; in fact, this very feature names an entire class of friction-based processes: the well-established solid-state welding [167].

While the idea of using frictional heat to additively manufacture metallic parts was based on general principles stemming from solid-state welding processes, one in particular provided the foundation for this novel AM approach: the Friction Surfacing (FS). Originally conceived in the 1940s and considered a variant of friction welding in the 1990s [166], this process consists of a rotating consumable stud that is brought into contact with a metallic substrate; an axial force is applied onto the stud, normal to the substrate, which results in friction between both surfaces. In turn, the friction generates heat, which increases the temperature at the tip of the stud. This region is softened as a consequence, forming a viscoplastic layer of stud material. At this point, either the substrate or the stud is moved along x and/or y directions. As a result, the softened tip is deformed and left deposited along the path taken by the stud and/or the substrate. Those process stages can be visualized in Fig. 2.15 [166].

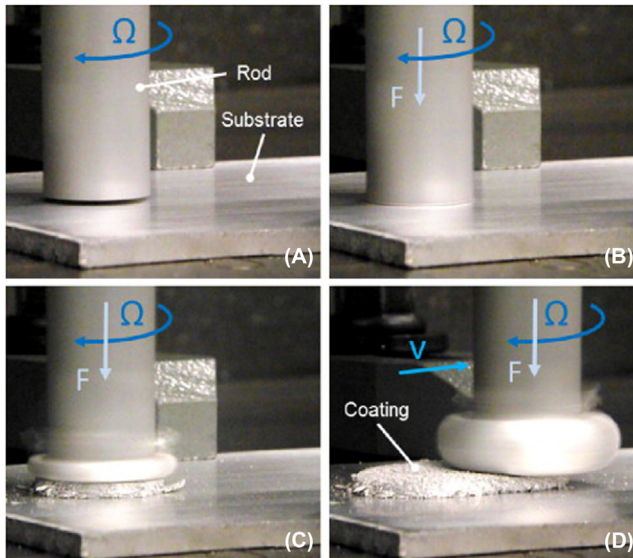


Figure 2.15 Stages of the Friction Surfacing process illustrated during the deposition of an AA6082-T6 stud onto an AA2024-T3 substrate. (A) The stud rotates at an angular velocity Ω , before establishing contact with the substrate; (B) rotating stud touches the substrate and an axial force F is applied; (C) the tip of the stud is softened; (D) the stud moves horizontally concerning the substrate at a speed v , leaving a coating of deposited stud material [166,168].

Source: From J. Gandra, et al., Friction surfacing—a review, J. Mater. Process. Technol. 214 (2014)1062–1093.

Originally, FS was envisaged as an alternative to produce hard coatings over metallic parts, which could enhance surface hardness, wear resistance [168–171], as well as act as a repairing technique for worn or cracked components [172,173]. However, given the process idiosyncrasies, its use as an alternative AM technique appears to be a natural step forward in the development cycle of the process. In this scenario, the stud acts as the feedstock material, and the FS would be programmed to deposit several layers stacked onto each other, akin to any other layer-by-layer AM process, yet without ever surpassing the melting temperature of the feedstock. The FS can be used as an AM process without further adaptations, as reported by some authors. Dilip et al. used multilayer depositions in combination with CNC machining to produce an AISI 410 martensitic steel block with fully enclosed cavities [174]. Shen et al. demonstrated the feasibility of the FS for AM using an AA5083 alloy as the feedstock material [175]. The authors observed that a 6-layer stack achieved a good homogeneity both in terms of grain size and hardness. However, one issue arising from using the FS as-is for AM is the formation of an excess of material that is not effectively deposited, which is commonly termed *flash* [176,177]. This excess normally ends up surrounding the consumable stud, representing a material loss during the process and consequently decreasing its efficiency. Thus, to suppress flash formation, an adaptation that has been vastly adopted both on the laboratory and industrial scales is the use of a rotating shoulder surrounding the consumable stud [178]. Made of a harder alloy with a higher melting temperature compared to the stud, this shoulder acts both increasing the total energy generated by friction and also pressing down the flash formed as the stud is consumed, increasing, therefore, the amount of stud material that is eventually deposited. The working principle of this adaptation is illustrated in Fig. 2.16 and is commonly referred to as Additive Friction Stir Deposition (AFSD) [178–180] or Manufacturing (AFSM) [181].

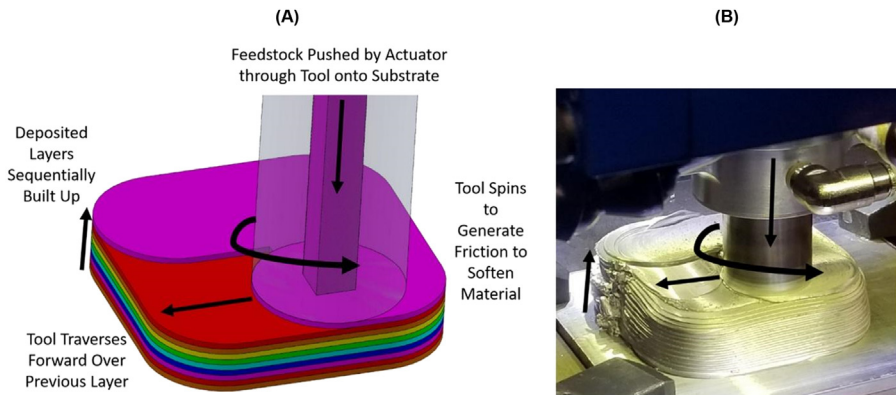


Figure 2.16 Schematics of the rotating shoulder encircling the consumable stud on the AFSD process [179].

Source: From G. Stubblefield, K. Fraser, B. Phillips, J. Jordon, P. Allison, A Meshfree computational framework for the numerical simulation of the solid-state additive manufacturing process, additive friction stir-deposition (AFS-D). Mater. Des. 202 (2021) 109514.

As a friction-based AM process, the AFSD currently finds itself at a higher development stage compared to the unadapted FS, which is mostly attributed to the advantages described earlier. The idea of adapting the FS for AM was initially patented in 2016 by the Meld Manufacturing Corp. (USA) [181–183]; since then, many authors studied the process with many different alloys, including copper [184], aluminum [185–188] and Inconel [186,189] ones. For those materials, in particular, the literature reports that the AFSD process promotes a general reduction in grain size in comparison to the consumable rods as received [185,188–190]. The fine, equiaxed grains were directly attributed to the dynamic recrystallization occurring during the AFSD process, triggered by the high shear rates and temperatures [185,186]. The authors indicate that this dynamic recrystallization of aluminum and Inconel alloys is most likely continuous, that is, without nucleation and growth stages, which can also occur in solid-state, friction-based welding processes [191,192]. Moreover, similar to the observations that were drawn from the unadapted FS, Rivera et al. found no discernible grain size gradients from top to bottom of a 6 mm thick AA2219 specimens manufactured by AFSD [185]. For Inconel 625 specimens, Avery et al. also reported a somewhat homogeneous microstructure along with the thickness; however, the layer interfaces showed an even greater grain refinement, reaching submicron diameters on average [186].

2.5 Ultrasonic metal additive manufacturing

This solid-state technique is capable of joining similar and dissimilar metal foils near room temperature by scrubbing them together under pressure and ultrasonic vibrations forming gapless, 3D metal parts [193]. Fig. 2.17 illustrates the ultrasonic additive manufacturing (UAM) equipment. The metal foil gets in contact with the sonotrode/horn subsequently being joined to the predeposited metallic base plate via high power ultrasonic vibrations. In reason of its circular format, the sonotrode

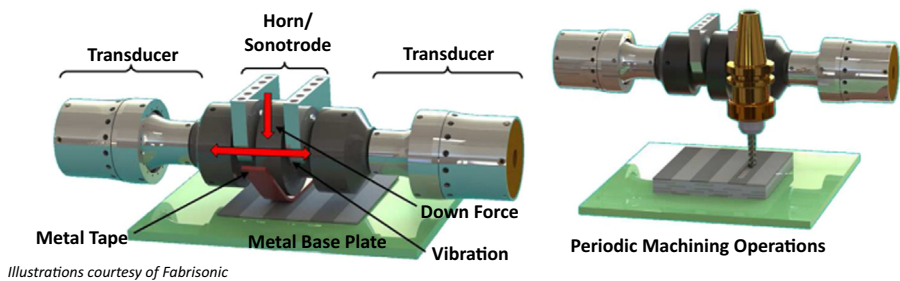


Figure 2.17 Schematic representation of UAM (left) and the subtractive CNC stage during the UAM process (right).

Source: From A. Hehr, M.J. Dapino, Dynamics of ultrasonic additive manufacturing, *Ultrasonics* 73 (2017) 49–66.

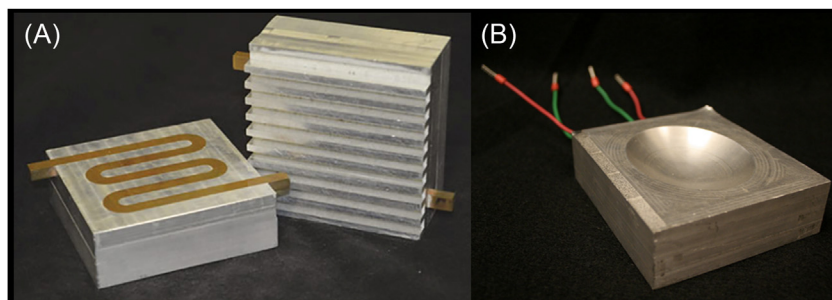


Figure 2.18 (A) heat transfer/cooling and (B) embedded electronic components fabricated by UAM.

Source: Fabrisonic.com.

performs a linear foil welding in a prescribed direction. The sonotrode coupled to high power piezoelectric transducers increases the delivered weld power substantially (from 1 to 9 kW), enabling strong and stiffer materials to be joint. In the meantime, periodic CNC machining might be employed aiming to create internal complex structures and net shaping the welded components [193,194].

The main applications of UAM are related to heat transfer devices and dissimilar materials joining, where aluminum and copper alloys as well as and mixed are easily joined since solidification is avoided and thus intermetallic formation is suppressed. Moreover, embedded electronics is benefited, where the inclusion of heat-sensitive components into the metal plates is possible. Both examples are illustrated in Fig. 2.18. Despite being unique, UAM suffers from limitations regarding design complexity and is not capable of producing parts without a CNC milling step. However, costs, the absence of thermal side effects, and the possibility of joining dissimilar materials still make UAM an attractive technique if compared to fused-based ones for applications such as electronics.

2.6 Artificial intelligence in additive manufacturing

2.6.1 Introduction

In the last two decades, *Design of Experiments* (DoE), an applied statistics branch, has been extensively used to conduct systematic experimental campaigns in joining technologies, and more recently in Additive Manufacturing (AM) processes. The preeminent objective is to obtain a combination of process-relevant parameters that, when optimized, yield an array of desired properties (e.g., mechanical, microstructural, etc.). Furthermore, by manipulating multiple inputs at the same time, DoE can identify important interactions, determining their effect on the desired output. However, the DoE approach usually involves trial and error, which is time-consuming and costly, particularly for metal AM [195–197].

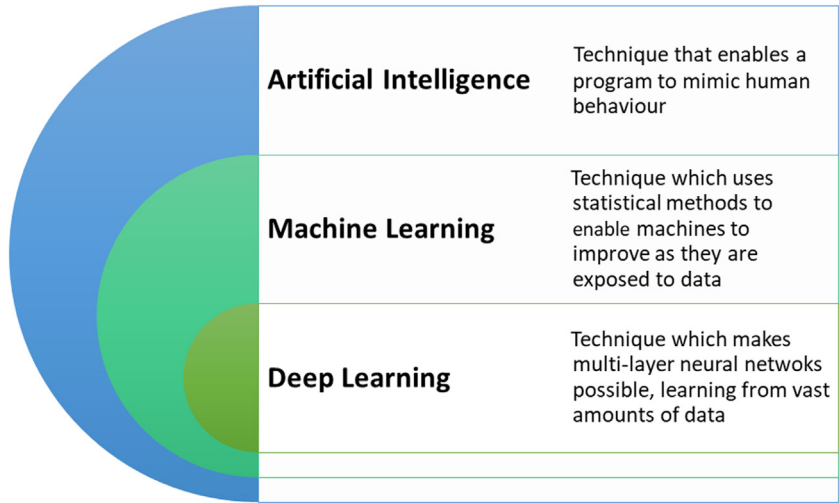


Figure 2.19 Relationship between AI, ML, and DL [201].

Source: From F. Rosenblatt, The perceptron: a probabilistic model for information storage and organization in the brain, Psychol. Rev. 65 (1958) 386–408.

Physical-based finite element analysis (FEA) can provide valuable insight into the underlying mechanism for the formation of specific features during processing, such as melt pool geometry, keyhole, and microstructure [198]. Nevertheless, sophisticated FEA techniques, for example, computational fluid dynamics, tend to focus on a single or limited number of tracks and layers, restricting the ability to predict mechanical properties of parts on a macroscale [198–200].

To tackle the aforementioned challenges, researchers are steadily exploiting the use potential of Artificial Intelligence (AI) techniques, namely Machine Learning (ML) and Deep Learning (DL) (see Fig. 2.19). In addition, the increase of data availability is enabling more tangible progress in the uptake of AI in the AM sector.

2.6.2 Learning methodology

The common ML workflow can be characterized by five core tasks [202], as follows in Fig. 2.20.

2.6.3 Machine learning

Machine learning (ML) is about designing algorithms that build upon the language of mathematics to automatically extract valuable information from data, that is, patterns and structure by optimizing the parameters of the model [203]. A model is said to learn from data if its performance on a given task improves after the data are taken into account, and robust if capable to reliably generalize for unseen data, which we may care about in the future.

 Get Data	The first step to construct an ML algorithm is to get the data. This can either be static or dynamic data, i.e., data from existing AM open-sourced libraries and repositories, or directly from sensor data acquisition of the printer.
 Clean, Prepare & Manipulate Data	This is the most crucial step in the process, and avoid incurring in the well known idea of "Garbage in, garbage out". Having a clean data set, exempt from missing, unorganised or noisy elements, ultimately helps with the model accuracy. The data is then split in training and test set, usually 70/30, respectively.
 Train Model	The data set connects to an algorithm, and the latter leverages sophisticated mathematical modelling to learn and develop predictions. Typically, algorithms fall into regression or classification categories.
 Test Data	The model should be validated against the test set defined in task 2. A careful analysis to statistical metrics should be carried out to assess the performance of the model when generalising to new observations.
 Improve	Should the model perform with unsatisfactory accuracy, the following actions might help in the refining process: 1) If available, use more data; 2) Reconsider the algorithm choice; 3) Adjust the algorithms hyperparameters.

Figure 2.20 Machine learning workflow.

Source: Based on Centric. Machine learning: a quick introduction and five core steps.

2.6.3.1 Regression

Numerous problems in engineering include investigating the relationship between two or more variables [204]. Such relationships can be captured and interpreted with the use of statistical techniques such as Regression analysis. For instance, in L-PBF, the density of a printed part is related to the scanning speed. Thus, regression analysis can be used to construct a model able of predicting the density for a given scanning speed, as well as the optimized speed that yields maximum density.

2.6.3.2 Linear and polynomial regressions

Linear and polynomial regressions are simple and useful algorithms that every engineer dealing with AM process design and optimization should carry in their portfolio, to establish meaningful physical interpretation between process parameters and target values.

2.6.3.3 Formulation

The general form for multivariate linear and polynomial regression can be expressed by Eqs. (2.1) and (2.2), respectively [205]:

$$y_i(x_1, x_2, \dots, x_n) = \beta_0 + \sum_{j=1}^n \beta_j x_{j,i} + \varepsilon_i, \quad (2.1)$$

$$\begin{aligned}
y_i = & \beta_0 + \sum_{j=1}^n \beta_j x_{j,i} + \sum_{j=1}^n \sum_{k=1}^n \beta_{jk} x_{j,i} x_{k,i} + \dots \\
& + \sum_{j=1}^n \sum_{k=1}^n \dots \sum_{p=1}^n \beta_{jk\dots p} x_{j,i} x_{k,i} \dots x_{p,i} + \varepsilon_i. \quad (2.2)
\end{aligned}$$

where $x_{j,i}$ corresponds to the i th observation on the n th independent variable; $x(j,i)x(k,i)$ relates to the quadratic effect of n th independent variable if $j = k$, and to the 1st-order interaction effect between variables j and k , $\forall j \neq k$. Additionally, β_0 corresponds to the intercept term, whereas $\beta_j, \beta_{jk}, \beta_{jk\dots p}$ are the regression coefficients. The far right term of Eq. (2.2) relates to higher p th order monomials. Writing Eq. (2.2) in matrix notation:

$$\vec{y} = \mathbf{X} \vec{\beta} + \vec{\varepsilon}. \quad (2.3)$$

The vector of estimated polynomial regression coefficients is obtained using Ordinary Least Squares (OLS) estimation [204], thus:

$$\hat{\vec{\beta}} = (\mathbf{X}^\top \mathbf{X})^{-1} \mathbf{X}^\top \vec{y}, \quad (2.4)$$

where $\mathbf{X}^\top \mathbf{X}$ is known as the normal matrix, and $\mathbf{X}^\top \vec{y}$ is the moment matrix of the regressors.

2.6.3.4 Gaussian process regression

Gaussian process regression (GPR) is a nonparametric, Bayesian approach that allows for reliable predictions on smaller data sets, allowing for uncertainty measurements on the predictions. GPR is not limited to the probability distribution specific function, but rather calculates it throughout all admissible functions that fit the data.

2.6.3.4.1 Formulation

Contrarily to well-known ML algorithms that learn exact parameters, the Bayesian approach infers a probability distribution over all possible values. Assuming a linear function of the type, a prior distribution on parameter is specified, and probabilities relocated based on observed data using Bayes' rule [206,207]:

$$p(w|y, X) = \frac{p(w|X, w)p(w)}{p(w|X)}, \quad (2.5)$$

where $p(w|y, X)$ refers to the distribution posterior, $p(w|X, w)$ to the likelihood, and $p(w|X)$ to the marginal likelihood. The predictive distribution can be computed as follows [206,207]:

$$p(f_*|x_*, y, X) = \int_w p(f_*|x_*, w)p(w|y, X)dw, \quad (2.6)$$

where x^* is a test observation, and f^* is the prediction associated to x^* . Since it is assumed that the prior and likelihood are Gaussian, then the predictive distribution is also Gaussian. Therefore, a prediction can be obtained using the mean, and variance to quantify the uncertainty.

In GPR, the prior is specified by the following [206,207]:

$$f(x) \sim GP(m(x), k(x, x')), \quad (2.7)$$

where $m(x)$ and $k(x, x')$ are the mean and covariance functions, respectively.

Although Eq. (2.9) can include noisy observations, this formulation will focus solely on noise-free observations. Extending Eq. (2.9) to a multivariate distribution, we obtain [206]:

$$\begin{bmatrix} f \\ f_* \end{bmatrix} \sim N\left(\begin{bmatrix} \mu \\ \mu_* \end{bmatrix}, \begin{bmatrix} K(X, X) & K(X, X_*) \\ K(X_*, X) & K(X_*, X_*) \end{bmatrix}\right). \quad (2.8)$$

If there are n training points and n_* test points, then $K(X, X_*)$ denotes the $n \times n_*$ matrix of the covariances evaluated at all pairs of training and test points. Similarly reasoning is applicable to the other entries of the last equation.

The covariance function, also known as kernel of X , models the covariance between each pair of. There are numerous kernels available, although the most popular is the composition of a constant and radial basis function (RBF) kernels [206,207].

$$k(x, x') = \sigma_f^2 \exp\left(\frac{1}{2l^2} \|x - x'\|^2\right), \quad (2.9)$$

where σ_f^2 are the signal variance and l the length scale. Finally, the noise-free predictive distribution is given by Rasmussen and Williams [206].

2.6.3.4.2 Applications

In the context of AM, GPR has been used for process design and parameter optimization. Some examples of applications of GPR in AM are summarized in Table 2.1.

Table 2.1 Applications of GPR within the scope of AM processes.

Process	Description	References
SLM	Material: SS316L Input: Laser power scanning speed Output: Melt pool depth	[208]
SLM	Material: SS17–4 PH Input: Laser power scanning speed Output: Porosity	[209]

2.6.4 Deep Learning

Deep learning (DL) is a subset of ML that uses multiple-layer neural networks (NN) to progressively extract higher-level features from the raw data. NNs attempt to replicate the behavior of the human brain through a combination of data inputs, weights, and biases, working together to accurately recognize, classify, and describe objects within the data. The difference between ML and DL relates to the degree of human intervention in the learning process of the algorithm; that is, if we were to analyze solidification defects, such as porosity and hot cracking, it would require human expertise in ML to determine the characteristics distinguishing both defects, whereas in DL this “hierarchization” is automatic. With that said, DL would require a larger amount of data to improve its accuracy [210].

2.6.4.1 Multilayer perceptrons

A multilayer perceptron (MLP) can be defined as a network of *neurons* known as *perceptrons*. This idea was introduced by Rosenblatt in 1958 [211], from which a single output is computed as a result of multiple real-valued inputs forming a linear combination according to its input weights and subjecting the output to an activation function.

2.6.4.2 Formulation

The mathematical formulation for a single perceptron is given by [212]:

$$y = \varphi \left(\sum_{i=1}^n w_i x_i + b \right) = \varphi (\mathbf{w}^\top \mathbf{x} + b), \quad (2.10)$$

where $\mathbf{w}$ denotes the vector of weights, $\mathbf{x}$ is the vector of inputs, b is the bias, and φ is the activation function. The signal-flow representation is depicted in Fig. 2.21.

Originally, the single perceptron used a Heaviside step function. However, nowadays, with the multiplicities of multilayer architecture, can take various forms, such as logistic sigmoid $1/(1 + e^{-x})$, hyperbolic tangent $\tanh(x)$, among others. Perceptrons should be used as building blocks to form the MLP architecture, capable of mapping the relations between input and outputs via hidden layers. By propagating the signal layer-by-layer, Fig. 2.21 can be expanded and generalized, where the input to the subsequent hidden layer is. The mathematical formulation of such a

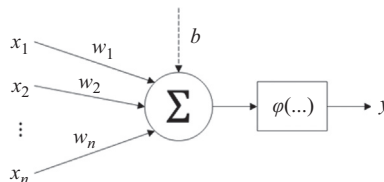


Figure 2.21 Single-layer perceptron signal-flow representation.

feedforward network with a single hidden layer with nonlinear activation functions and a linear output layer can be written as [212]:

$$\mathbf{x} = \mathbf{f}(\mathbf{s}) = \mathbf{B}\varphi(\mathbf{A}\mathbf{s} + \mathbf{a}) + \mathbf{b}, \quad (2.11)$$

where $\mathbf{s}$ and $\mathbf{x}$ correspond to the input and output vector, respectively; $\mathbf{A}$ and $\mathbf{B}$ are the weight matrixes of the first and second layers, correspondingly; $\mathbf{a}$ and $\mathbf{b}$ are the bias vectors of the first and second layers, respectively. The function φ represents the elementwise nonlinear activation. MLP is typically used for supervised learning problems from which the model has to work out the dependency between input and outputs from a training set. Hence, the aforementioned weights and biases have to be adjusted to an optimal value for given training pairs $(s(i), x(i))$ according to the squared reconstruction error criterion [212]:

$$SRE = \sum_{i=1}^n \|\mathbf{f}(\mathbf{s}(i)) - \mathbf{x}(i)\|^2. \quad (2.12)$$

The adjustment of weights and bias is performed using the backpropagation algorithm, where the partial derivatives of the cost function Eq. (2.9) concerning the different parameters are propagated back through the network.

2.6.4.3 Applications

MLP is extensively used for parameter optimization and in situ monitoring of AM processes. Table 2.2 summarizes some of the works performed to date in

Table 2.2 Applications of MLP within the scope of AM processes.

Process	Description	References
SLM	Material: Ti-6Al-4V Inputs: Spreader translation and rotation speed Outputs: Powder bed roughness, spread speed	[213]
SLM	Material: SS304 Inputs: Acoustic signals Outputs: Classify melting states	[214]
SLM	Material: In625 Inputs: Intensity, morphology, thermal profile of melt pools Outputs: Distinguish between the overhang and bulk build states	[215]
SLS	Material: PLA Inputs: Layer thickness, laser power, feed rate Outputs: Open porosity	[216]
DED	Material: Cu-coated steel wire Inputs: Wire feed rate, welding speed, arc voltage, nozzle-to-plate distance Outputs: Bead width, height	[217]

terms of the input data and desired outputs in metal AM processes, subjected to different materials.

2.6.5 Future trends in AI for AM

The majority of research endeavors involving the linkage of AI with AM focus on the engineering and design aspects of AM [198]. Thus, it is crucial to translate state-of-the-art ML and DL techniques to tackle the following relevant topics in AM.

2.6.5.1 Topology optimization

The use of AI in topology optimization (TO) is still limited. Although research has been carried out to generate topologically optimized designs via DL, these mostly relate to 2D structures [198]. Nonetheless, efforts are being carried out to accelerate the use of DL in 3D and determine the optimal computational strategy for its deployment. Promising work was carried out by Banga et al. [218] using 3D encoder-decoder convolutional neural networks (CNN), having achieved an overall reduction in computational time of 40% while attaining accuracies in the order of 96%. With the steady advancement of ML and DL algorithms and growing 3D TO data, the leap from 2D to 3D will be achieved soon.

2.6.5.2 Microstructural characterization

Due to the melting and solidification nature of metal AM, it is crucial to evaluate and quantify the microstructure characteristics, as well as solidification defects that might arise as a function of the process parameters. Assessing the resulting microstructure solely with experimental campaigns is tedious and costly. Although the data volume to justify the use of a DL must be sufficiently large (e.g., 1000 images per cycle), transfer learning can be used to migrate parameters previously trained to a newly developed DL algorithm. Additionally, data augmentation can help improve accuracy and reduce the data size (e.g., random cropping, flipping, mirroring, warping). Once representative data set is at hand (either real or virtually created), conditional Generative Adversarial Network (cGAN) is a suitable algorithm to predict microstructure when conditioned to the process parameters.

2.6.5.3 Hybrid modeling

ML methods often work as black boxes and do not provide insight into the physics underlying the process. To this end, a hybrid approach combining physical models with ML preserves physical information, while still improving the flexibility and precision of the model. This approach requires a combination of strong domain knowledge and ML expertise, resulting in robust and physically interpretable models [219] Fig. 2.22 depicts schematically the principle underlying hybrid modeling and its potential in minimizing estimation error by combining a fundamental and data-driven

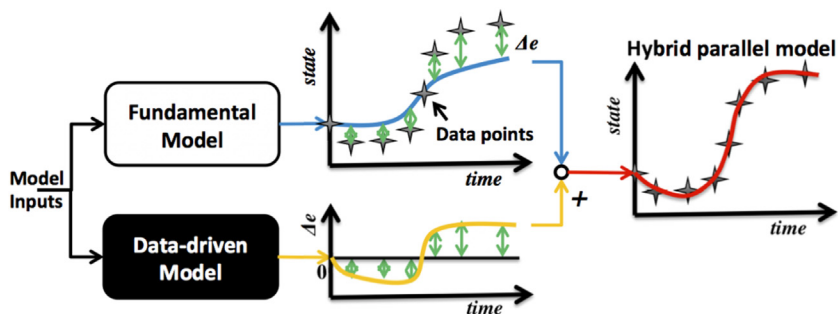


Figure 2.22 Schematic principle of hybrid modeling.

model. This type of model is of great significance in AM when it comes to advanced process monitoring, optimization, and control. One of many possible applications would be to characterize and control the transient heat transfer evolution in the powder bed to avoid undesired sintering of the powder.

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Current trends of metal additive manufacturing in the defense, automobile, and aerospace industries

3

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3.1 Introduction

Additive manufacturing (AM) has attracted much interest from industry and academia because of several potential improvements to production systems, such as waste material reduction, shorter manufacturing lead times, high flexibility, the feasibility of complex geometry products, and a shorter product development cycle. The demand for highly personalized objects, combined with the emergence of new business models and leaner supply chains, fuels interest in additive manufacturing technology [1,2]. In the last two decades, the use of additive manufacturing (AM) has exploded in various industries. Aerospace has gotten the most attention because of the major aircraft corporations' substantial investment in developing AM industrial applications. However, numerous studies have been conducted to make it a more adaptable and safe technology, which necessitates the development of novel materials, technologies, process design, and cost-effectiveness. High-cost and lead-time reductions, novel materials and unique design solutions, mass reduction of components through highly efficient and lightweight designs, and consolidation of multiple components for performance enhancement or risk management, for example, through internal cooling features in thermally loaded components or by eliminating traditional joining processes, are all real opportunities for metal additive manufacturing in aerospace applications. These advancements are being commercialized in various high-profile aerospace applications, including liquid-fuel rocket engines, propellant tanks, satellite components, heat exchangers, turbomachinery, valves, and legacy system maintenance [3].

Functional performance, lead-time reduction, lightweight, complexity, cost management, and sustainment are all technical and economic objectives that must be met in the aircraft industry. Each of these goals has a strong relationship with the others; thus, concerns from each component must be carefully addressed when choosing the best design solution. The relative relevance of these goals varies according to the aerospace application [4]. High-performance materials are utilized

with increasingly complicated designs in the quest to increase efficiency continually through cost, lead-time reduction, and attempts to reduce the bulk of flight components. This must be done at a reasonable cost and on time to meet retail orders or mission criteria. Traditional manufacturing technologies and tactics have been established over decades to meet these aerospace design objectives for various application types; however, additive manufacturing is having and will continue to have a significant impact on design and manufacture. It is feasible to optimize the material distribution while maintaining the component's mechanical and other performance criteria by exploiting the design freedom of metal AM. Components can also be combined to reduce risk and cost for several components while also lowering potential failure modes across joints. Using mechanical, thermal, and other optimization methodologies to design complicated parts that were previously impossible to manufacture, such as conformal cooling channels on combustion chambers or turbine blades, better performance (above that of conventional production) is also achievable [5,6]. While the current main motivation for using AM in aerospace applications is the reduction in lead times, specific manufacturing scenarios give AM advantages over traditional manufacturing.

This study includes a detailed assessment of metal AM applications in the aerospace sector, as well as a current state-of-the-art review. By presenting these successful examples, the benefits of additive manufacturing and its obstacles in aerospace are carefully recorded, supporting the use of additive manufacturing in this sector while considering all of the constraints and requirements.

3.2 Metal additive manufacturing systems

Each AM technique has its own set of features regarding material that may be used: processing methods, and capabilities. Despite this, most of them use a pointwise approach and metal powder as a raw material. Metals are common structural materials that have been widely produced through conventional production procedures (such as casting, forging, and welding). By producing complex structures, lowering the number of required structural components and production costs, and shortening the processing timeframe, AM can increase the use of metals. As a result, AM can alter the current lifecycles of metal parts. We must investigate novel materials, techniques, and architectures to use the benefits of 3D printing in practical applications fully. Fig. 3.1 shows how AM technologies for metallic materials can be classified based on the type of feedstock materials and energy source used. Metal AM technologies frequently use powder and wire feedstock materials. Powder bed fusion (PBF) (Fig. 3.1A) and directed energy deposition (DED) (Fig. 3.1B) are the most common metal AM technologies that utilize powder as a feedstock material. SLM (selective laser melting) and SLS (selective laser sintering) are two PBF processes that use the laser as an energy source. Optical fiber lasers, rather than CO₂ or Nd:YAG lasers, are used in current laser-based PBF systems, enhancing the laser's consistency and power. Electron beam melting (EBM) is another PBF

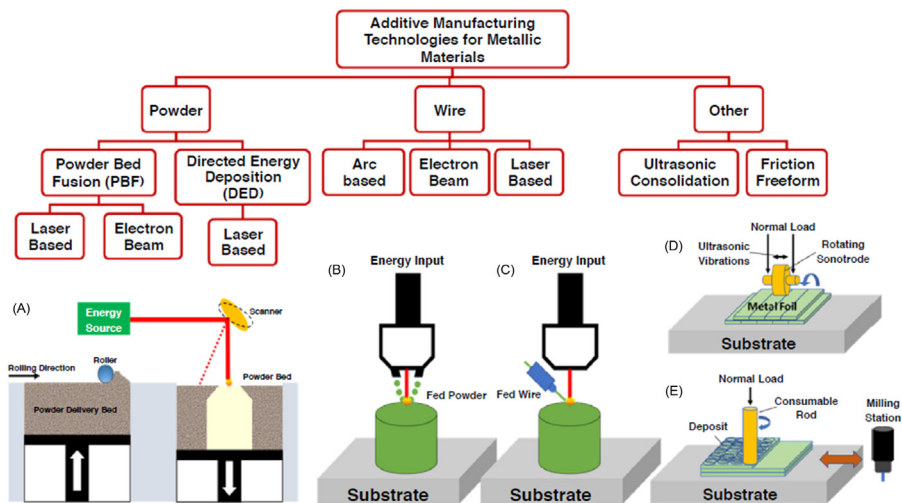


Figure 3.1 Classification of metal additive manufacturing techniques: (A) powder bed fusion (PBF); (B) directed energy deposition (DED); (C) wire-based deposition; (D) ultrasonic consolidation; and (E) friction freeform [7].

technology that uses a high-power electron beam as the energy input instead of a laser. EBM parts are created in a vacuum room, unlike laser-based PBF processing, which requires an inert gaseous printing environment. Before each layer is printed, the electron beam preheats the entire powder bed in EBM production. This could help eliminate residual stresses in the produced product and create a martensitic phase as a result of rapid cooling. The most recent PBF systems can achieve powder layer thicknesses as low as 20 mm and feature sizes as small as 100–150 mm [8]. The fine resolution could significantly increase the density and quality of as-fabricated parts and the surface finish. The quad-laser system is another sophisticated configuration of modern SLM machines that significantly increases print rate [9]. Directed energy deposition (DED) is a metal AM process that uses a carrier gas to feed the powder(s) directly to the laser's focal point. When the laser scans the melted region's surface, the prior molten pool solidifies quickly, forming a bulk structure. Optical fiber lasers are used as an energy input in modern DED equipment to increase product quality and dependability.

3.3 AM materials for aerospace applications

Aluminum alloys, stainless steel, titanium alloys, nickel- and iron-based superalloys, copper alloys, cobalt alloys, refractory alloys, and steels, among other metallic materials, are unique materials for AM in aerospace. Most of them are utilized in prealloyed powder form, which is often created by gas atomization, or in wire form, depending on the procedure [10]. Since the beginning of the aerospace

industry, aluminum alloys have been a critical component. Aluminum was the most widely used material in aerospace until recent breakthroughs in composite technologies [11,12]. Its low cost, lightweight, high strength-to-weight ratio, and ease of manufacture made it the most widely used material. However, aluminum alloys have poorly raised temperature capabilities, and corrosion resistance in some high strength aluminum alloys is weak, restricting their applicability.

Due to their high specific strength, good corrosion resistance, and high-temperature stability, titanium alloys have piqued interest in aircraft applications [13,14]. Polymer matrix carbon fiber composites (PMCs), widely utilized in current aeroplanes, are also electrochemically compatible with titanium alloys. Because of their great thermal stability and particular strength, titanium alloys are widely used in aircraft. Titanium alloys are also suitable for cryogenic applications, such as rocket propellant tanks, because they do not undergo a ductile-brittle transition at low temperatures [15].

Superalloys based on nickel and iron have become important materials for producing disks and blades in high-pressure turbines for gas turbine engines. They are also employed in valves, turbomachinery, injectors, igniters, and manifolds, among other high-temperature and cryogenic applications [16,17]. Their superior mechanical qualities under intense temperatures, high pressures, and corrosive conditions significantly improve the efficiency of current aircraft engines [18]. Nickel-based superalloys now account for more than half of the mass of sophisticated aviation engines [19]. High-pressure hydrogen applications, such as rocket engines often use iron-based superalloys to reduce hydrogen environment embrittlement [20].

Refractory elements like niobium, tantalum, tungsten, and alloys like C-103 [21] are other metal alloys that can be employed in aerospace applications. These are employed in applications that need severe temperatures, such as in-space radiatively cooled rockets. Other materials, such as cobalt-based alloys like Co-Cr and Stellite, are also employed in high-temperature applications [22,23]. Many of these materials are used as feedstock for metal AM machines, and there have been varying levels of research into the material characteristics and properties produced using AM methods, with demonstrations of mechanical properties that are comparable to, if not better than, those associated with traditional manufacturing methods [24–26]. The process parameters and postprocessing operations must be tuned to reduce porosity, residual stress, and fracture propensity and include improved postprocessing processes as needed to improve material properties, such as optimized heat treatments and HIP. Table 3.1 lists the most common AM materials used in aircraft.

3.4 Aerospace applications of AM

Complex geometries are common in aerospace components, typically comprised advanced materials such as titanium alloys, nickel superalloys, special steels, or ultrahigh-temperature ceramics, which are difficult, expensive, and time-consuming to produce. Furthermore, aircraft production runs are often small, ranging from a

Table 3.1 Popular additive manufacturing (AM) materials used for aerospace applications [27].

Al base	Fe base	Ti base	Ni base	Cu base	Refractory	Co base
AlSi10Mg 7050 6061 4047 2024 A205 F357	SS 15-5 GP1 SS 17-4PH SS 420 SS 304L SS 316L Tool Steel (4140/4340) SS347	Ti6Al4V Ti-6-2-4-2 γ -TiAl	Monel K-500 Hastelloy-X C-276 Inconel 625 Inconel 718 Waspalloy Haynes 230	CU110 C18150 C18200 GRCop-42 GRCop-84 Glidcop	Ta Mo W C-103	CoCr Haynes 188 Stellite 21

few thousand to several million pieces. As a result, AM technology is particularly well suited to aircraft applications. The potential applications of AM in the aerospace industry are described in Fig. 3.2. The specific applications of AM processed parts in aerospace applications are discussed in the subsequent sections.

The term “space industry” describes commercial operations, including orbital launch vehicles, manufactured components that travel beyond Earth’s orbit, and other related services [28]. To continue to drive exploration and new missions, the space industry has often relied on new, advanced technologies. Early AM uses for space applications demonstrated potential advantages in design and production, as

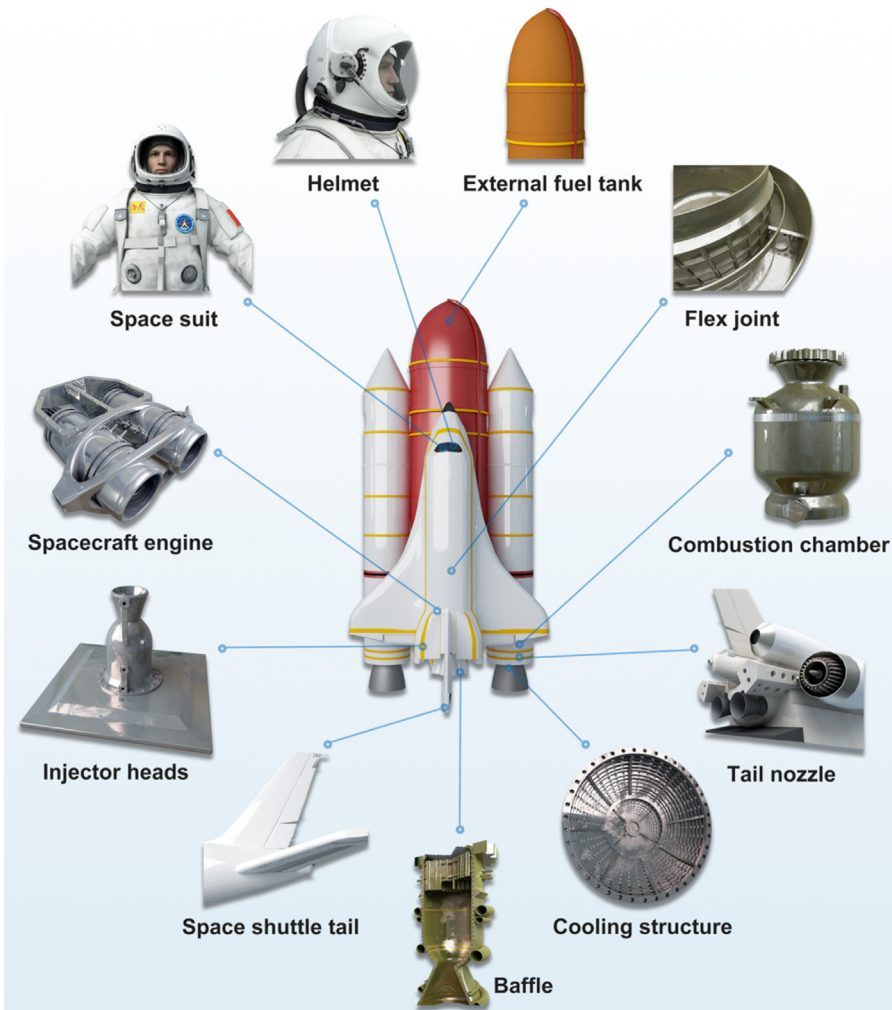


Figure 3.2 The current and potential application of additive manufacturing in aerospace industry.

mentioned in [29]. Since then, AM technology has progressed, and confidence in metal AM has grown dramatically throughout the aerospace sector, resulting in AM playing a larger part in the fabrication of components for launch vehicles and satellites [30]. Both the Sentinel-1A and Sentinel-1B, released in 2014 and 2016, used traditional manufacturing techniques, allowing a set of new brackets built by RUAG and Altair and made using AM techniques to be compared to the traditional design. RUAG designed and built an optimized version of an existing Antenna bracket depicted in Fig. 3.3 in collaboration with EOS and Altair [31,32].

A turbine housing produced of stainless steel utilizing the hybrid manufacturing technique is shown in Fig. 3.4. The turbine housing is 180×150 mm in size, with a build-up period of 230 minutes for deposition and 76 minutes for milling. Combining the precision accuracy of CNC machining with the freeform construction capacity of the AM process, this hybrid manufacturing technique creates high-quality finished products.

The General Electric LEAP engine fuel nozzle is probably the most well-known AM component application in aircraft. Since its inception in 2015, approximately 30,000 nozzles have been made, and it is still in production. The fuel nozzle,

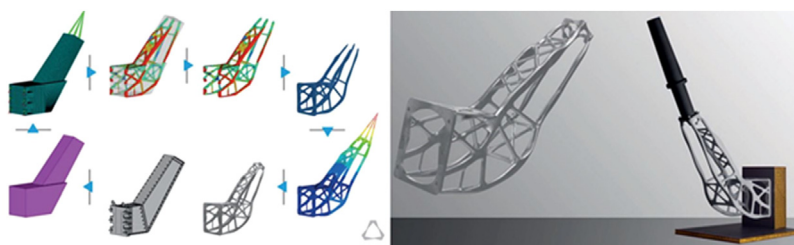


Figure 3.3 Topology optimized AM processed antenna bracket for satellite [33].

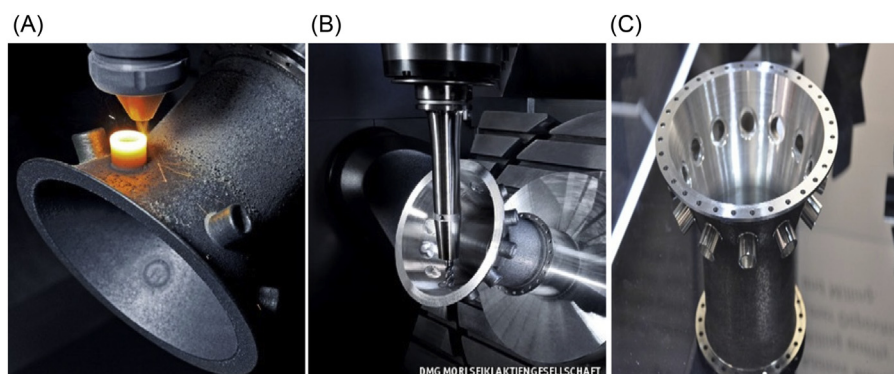


Figure 3.4 Turbine housing fabricated by the LASERTEC 65 3D System using (A) multi-axis deposition and (B) multi-axis machining. (C) Finished part.

Source: DMG Mori and Siemens.



Figure 3.5 The GE LEAP fuel nozzle [34].

Source: General Electric, New manufacturing milestone: 30,000 additive fuel nozzles, GE Additive, 2018. <https://www.ge.com/additive/stories/newmanufacturing-milestone-30000-additive-fuel-nozzles%0Ahttps://www.ge.com/additive/blog/new-manufacturing-milestone-30000-additive-fuelnozzles>.

illustrated in Fig. 3.5, is made of L-PBF and a Cobalt-Chrome alloy and is used on various commercial aviation engines.

There are examples from academia and commercial uses in the aerospace industry. Liou et al. [35] devised a hybrid method that combines multi-axis laser deposition with CNC machining. For freeform fabrication, the hybrid method gives improved build capabilities in terms of precision and surface quality, and it has been successfully employed to produce and repair functional metallic parts. The LMD technique was used to directly produce metallic components by Xue and Islam [36] from the Integrated Manufacturing Technologies Institute of the National Research Council of Canada. The materials investigated included nickel-based IN-625 and IN-738 superalloys, titanium-based Ti-6Al-4V alloy, cobalt-based Stellite 6 alloy, and iron-based CPM-9V tool steel. Mechanical tests revealed that LMD-processed materials are equivalent to, in some cases, better than traditionally cast and wrought materials. The LMD technique was successfully employed to manufacture net-shape Ti-6Al-4V airfoils with incorporated cooling channels, as shown in Fig. 3.6, which perfectly replicate the geometric features indicated in the computer-assisted design (CAD) model.

EOS' metal PBF machine is used by GE Aviation [37] to make next-generation jet engine components utilizing the direct metal laser sintering technique. The new gasoline nozzle seen in Fig. 3.7 is the AM-fabricated element that receives the most attention. Each LEAP engine contains 19 3DP-created fuel nozzles. Compared to the predecessor part created by traditional manufacturing, the new design has more

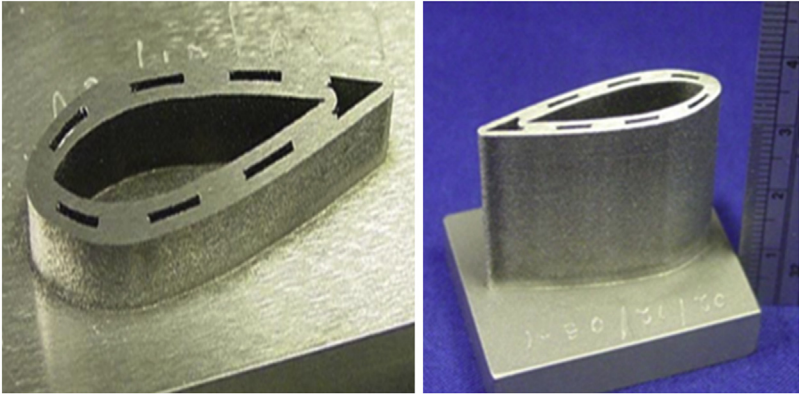


Figure 3.6 Ti-6Al-4V airfoils with embedded cooling channels built using the laser metal deposition process.

Source: Integrated Manufacturing Technologies Institute of the National Research Council, Canada.



Figure 3.7 GE LEAP Engine fuel nozzle created using direct metal laser melting via the EOS metal powder bed fusion machine.

Source: GE Aviation and EOS.

intricate cooling routes and support ligaments, resulting in a fivefold increase in service life. Engineers can employ a simpler design with the AM process, reducing the number of brazes and welds from 25 to only 5 and the number of pieces necessary to build the nozzle from 18 to 1. Furthermore, the weight of these nozzles is 25% less than that of the previous section.

3.5 Challenges and future prospectus of metal AM in aerospace industry

As evidenced by the applications discussed in the preceding sections, metal AM technologies provide much potential for technical and commercial advantages in aircraft applications. Despite the technological and financial benefits, these technologies face different obstacles in aircraft applications. This section discusses some of the present key obstacles and some potential future opportunities.

3.5.1 Challenges of AM in aerospace applications

3.5.1.1 Certification and standards

The lack of technical standards and certification techniques for metal AM in the aerospace sector, which has resulted from the rapid proliferation of AM technology over the last decade, is a key contemporary concern. The reproducibility, dependability, and quality of AM components for aerospace applications, these standards must be developed and agreed upon across the industry.

3.5.1.2 Structural integrity

For mission-critical aerospace applications, structural integrity is crucial, including dynamic loading in both high and low cycle modes, thermal cycling, and impact loading. In AM aerospace applications, fatigue response to dynamic loading is significant. Metallic AM-built components have relatively well-studied static mechanical characteristics, including hardness and strength that match or exceed traditionally created materials [24–26,38]. Dynamic mechanical qualities like fatigue and creep, on the other hand, have received less attention, and there is still a shortage of test data reporting among aerospace businesses [10].

3.5.1.3 Design for AM

DfAM (Design for Additive Production) can be utilized to optimize designs for the greatest manufacturing quality while minimizing support structures and postprocessing requirements [39]. This can also be used in conjunction with build simulations to help establish the best orientation for the part on the build platform, construction techniques to reduce residual stresses and resultant distortion, and support structure minimization. Simulations and DfAM processes take time and are dependent on the

AM engineer's engineering competence and the computational capability of the computer employed, but they may be necessary.

3.5.1.4 *Material characteristics*

For the aerospace industry, AM methods offer the distinct benefit of manufacturing difficult-to-machine materials such as titanium alloys, nickel alloys, specific steels, and ceramics. However, there is no standard material characteristics database in the AM business that provides mechanical attributes fabricated by diverse AM techniques, such as specifications of available materials' mechanical properties and more detail on how components made from these materials operate [12]. Engineers and designers cannot design without knowing everything there is to know about the materials utilized to make the things they are working on. Designers will not consider AM as a production process if the attributes for AM materials are not accessible [40].

3.5.1.5 *Process control*

The ability to fabricate in a predictable, repeatable, consistent, and uniform manner is critical for the AM process to be widely accepted by the industry. For each material and process, in-process monitoring, closed-loop feedback, and modeling and integrating process—structure—property connections with CAD/E/M tools are required. In situ sensors, particularly in temperature control, should be investigated to provide nondestructive evaluation and early defect identification. Improved process controls may also lead to less downtime, which is a big concern for many machines and processes right now [33].

3.5.2 *Potential future applications of AM in aerospace*

Novel custom alloys, bimetallic and multimetallic processing and characterization, extensive process—structure—property understanding, databases for AM materials, process certification, design optimization, and process simulations are significant future growth areas. Topology optimization and lattice structures can reduce mass, which is a demand in the aerospace industry. In both cases, improved components with increasingly complicated designs result from a better understanding of such structures' design and successful production. Further, shortly, the aircraft sector is projected to see the following potential applications of AM:

- AM for the design of aircraft components
- Fabrication of large-scale aerospace components using AM
- Functionally graded materials for aerospace applications

The key advantages of metal AM in aerospace, as mentioned below, are cost and lead-time reduction. With optimized design or the use of various alloys, mass reduction is also a huge opportunity area, but these techniques must be well understood and attribute well characterized. Furthermore, portion consolidation is a significant benefit in

this market. Metal AM's inherent part complexity capabilities enable high complexity within components, including internal features like channels and large surface areas for heat transfer applications. AM has now been demonstrated in several large-scale applications; thus, scalability is no longer an issue. These and other advantages of metal AM in aerospace offer much promise for widespread use, which will push the remaining hurdles to be addressed even further.

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Review of Microstructure and Mechanical properties of materials manufactured by direct energy deposition

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4.1 Introduction

Additive manufacturing (AM), a relatively latest technology of component fabrication, has gained extensive interest and curiosity in the manufacturing sector since last couple of decades. AM is primarily defined as the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies (ASTM) [1]. The actual definition refers to a computer-aided design model will be used in the component build process. For defining the build path of 3-dimensional deposition, some derivatives of traditional welding processes use a computer numerical controlled program or similar tools. Manufacturing with minimal tooling is an important characteristic for engineering industries as it saves both time and money.

The processing of powder materials from the feedstock by a laser energy source is called “Laser-assisted directed energy deposition” or DED, which has gained lot of interest in the recent past, and it is one of the most important metal AM processes to date. The direct energy deposition (DED) technology has been used in the processing of aerospace alloys for nearly last couple of decades. For example, the primitive applications were mostly for cladding, coating, and surface alloying. The update of efficient laser processing technologies with higher power input emerged the selective laser sintering with near net shape forming during 1990s [2].

4.2 Direct energy deposition

In direct energy deposition (DED), the component to be built needs to be constructed through design software, and further it will be sliced through the computer aided manufacturing (CAM) software such as “Master CAM.” Usually the tool

path, layer thickness, scan strategy, etc. will be adjusted and verified on the computer through the software interface. Then the verified sliced software file loaded on to the DED machine. Once again, the software will be checked for the tool path on the machine to ensure any abnormality for safe working.

Then, the process starts by depositing the predetermined powder material fed through the nozzle which forms the vertex and the energy source (say laser beam) will be impinging at the vertex to melt the material onto a build plate, where it solidifies and fuses materials together to build a structure layer by layer. In most DED machines, the nozzle is positioned on a multiaxis arm that can move in many directions, allowing for varied deposition. The procedure is usually carried out in a controlled environment with low oxygen levels. When working with reactive metals, electron beam-based systems operate in a vacuum, whereas laser-based systems operate in a totally inert chamber. In some cases, during metal deposition in open atmosphere, a shielding gas can also be used to cover the part and to avoid contamination. When a powder or wire is put onto the surface of an object, DED uses a heat source to melt it. While powder allows for more precise deposition, wire is more yielding in terms of material usage. To construct new features, the material is added layer by layer and solidifies from the melt pool. The thickness of the layers is normally 0.25–0.5 mm. Material solidification rates are extremely rapid, ranging from 1000°C to 5000°C per second [3]. Although overlapping and remelting the previous layers are inevitable and promote remelting and thus results in a uniform yet alternating layered microstructure, the cooling time has significant impact on the final microstructure and grain orientation.

4.3 Advantages and disadvantages

In DED, there is an ability to control the grain structure and therefore it allows us to repair of higher quality functional parts such as gas turbine components and other engineering components. Moreover in DED by regulating the deposition speed, the productivity can be enhanced, however at the expense of quality, and thus there is a compromise between speed and accuracy is needed. The larger volume components can be built in DED with minimal tooling. The DED process also allows us to build hybrid components with the combination of different materials as well as hybrid manufacturing such that both additive and subtractive processing in the same machine.

The disadvantageous of DED such as, depending on the material used, the finished product may require some postprocessing to produce the intended performance. Further in DED, there is limited materials for application and these technologies require additional research before becoming common [4,5].

4.4 Applications in different fields

Although Directed Energy Deposition mainly used to build things, it is more commonly utilized to repair or add material to existing components. The most common

applications of DED are to produce near-net-shape and rapid prototyping of metallic components, manufacture of complex geometries or customized builds may be with little or no machining, further to enhance the existing feature of any part/component, and, the last but not the least, is to repair and refurbishment of engineering components, predominantly in gas turbine components repair [4,5].

4.5 Microstructure and mechanical properties of different materials

This section will describe about the solidification microstructure and mechanical properties of various materials manufactured through DED

4.5.1 Steels

4.5.1.1 *Influence of powder characteristics on direct energy deposition process*

The powder characteristics having an influence on the DED process parameters and further, it may impact the final part properties as well as microstructure. For example, powder catchment efficiency and surface roughness are few among them. Didier Boisselier et al. studied different batches of SS316L powder and understood that in one of the powder materials, due to excessive carbon and oxygen ratios, it does not match the chemical composition. However, noticed certain changes in the microstructures and the presence of slags stuck between the layers. Similarly, in the other batch, the particle size distribution affected the powder flowability; therefore, the jet was not stable at the Nozzle orifice, which was affecting the component built and stability. Moreover, the porosity in the powder also influenced the quality of the component. In another batch, the powder had better catchment efficiency that resulted in better metallurgical quality as well as the good surface finish without any qualitative defects.

It is understood that the particle size distribution is important and may affect the powder flowability and the variation in chemical composition may result in the impurities. Also, the porosity in the powder material will result in the defects in the part to be built by DED. Therefore, while choosing the powder materials for DED, one must ensure to check the quality of the powder before printing as they may significantly influence the final part properties [6].

4.5.1.2 *Effect of laser rescanning strategy on the microstructure and mechanical properties*

The laser scanning strategy is an important aspect in the DED process; by altering or rescanning, there is a possibility to enhance the mechanical properties of a given material. Tae Hwan Kim et al. observed that after deposition of each layer by additional laser rescanning on the same layer, the yield strength and ultimate tensile

strength can be improved 1.2 times without compromising or the better ductility of the SS316L steel.

It is understood that the laser rescanning remelts the direct energy deposited layers and shallow in shape due to the change in the heat transfer behavior. Further, it is also noted that the laser rescanning suppresses the delamination cracking as opposed to simple laser energy deposition, which therefore enhances the final mechanical properties of the component manufactured. Also, the rapid solidification rates during the laser rescanning exhibits finer interlayers and therefore the better mechanical properties [7]. Figures 4.1 and 4.2 shows the DED processed microstructure and mechanical properties of SS316L steel.

4.5.1.3 Microstructure and mechanical properties of different steels

DED having excellent potential for production of complex geometries and when it comes to SS316L steel, the process parameters such as laser power, scan speed, powder feed rate, inert atmosphere, and scan strategy play a key role. The most important thing is that the control of the microstructure determines the mechanical properties of 316 L steel. However, the thermal history, solidification rate, and temperature gradient influence the final microstructure of the SS316L steel. For

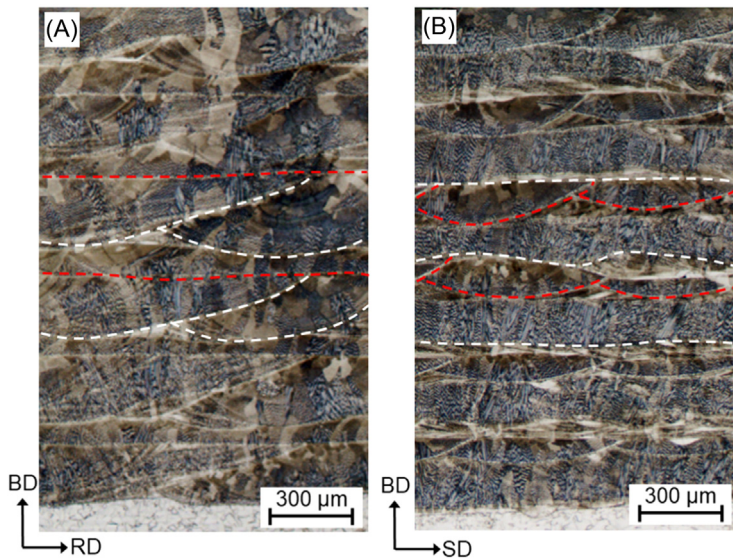


Figure 4.1 Representative optical images of SS316L rescanning (Res) samples: (A) scan direction and (B) rescanning direction.

Source: From T.H. Kim, G.Y. Baek, J.B. Jeon, K.Y. Lee, D.-S. Shim, W. Lee, Effect of laser rescanning on microstructure and mechanical properties of direct energy deposited AISI 316L stainless steel, *Surf. Coat. Technol.* 405 (2021) 126540.

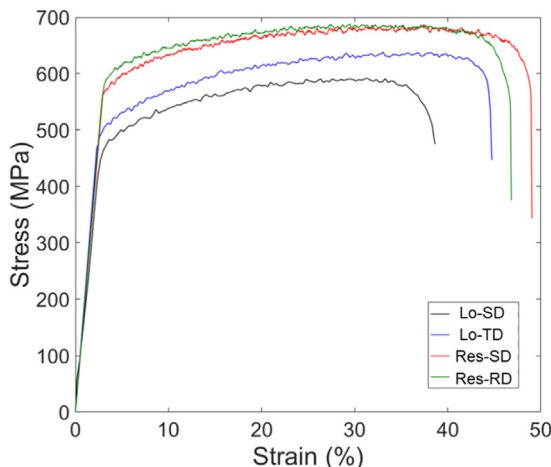


Figure 4.2 Representative tensile stress versus strain curves for the SS316L sample direct energy deposited (Lo) and rescanning (Res) samples (SD, scan direction; TD, transverse direction; RD, rescanning direction).

Source: From T.H. Kim, G.Y. Baek, J.B. Jeon, K.Y. Lee, D.-S. Shim, W. Lee, Effect of laser rescanning on microstructure and mechanical properties of direct energy deposited AISI 316L stainless steel, *Surf. Coat. Technol.* 405 (2021) 126540.

instance, the rapid solidification of DED processed builds results in very fine dendritic structure and correlates to better mechanical properties due to increase in dislocation density. On the other hand, the rapid solidification nature during the build process would also generate significant residual stresses in the steel; however, this can be eliminated by postprocessing techniques such as stress relieve annealing treatment. In SS316 steels sometimes, the oxide formation during DED may affect the final mechanical properties. Moreover, the anisotropy in the mechanical properties may be attributed the different directions of the DED built components, however can be minimized by heat treatment techniques. Sometimes it is observed that the recycling of powder feedstock significantly influences the quality of the build as well as final mechanical properties.

Reduced activation steel is one of the prominent creep- and radiation-resistant materials due to its radiation and high temperature and creep resistance. Therefore, it is mostly used in gas turbine as well as in nuclear power generation applications.

The DED processed reduced activation steel microstructure consists of high-density dislocations with fine martensite laths in the prior austenitic grains. The higher yield and ultimate tensile strength with better ductility and is corroborated to the microstructural features of this DED processed steel. With the quenching and tempering, heat treatment of the DED-processed reduced activation steel exhibits excellent ductility and significant improvement in the impact strength, however at the expense of reduced yield and ultimate tensile strength [8].

4.5.1.4 Process parameters influence on functionally graded steels by direct energy deposition

It is well known fact that manufacturing of functionally graded materials is one of the obvious advantages of DED. SS316 and Fe functionally graded material has been studied by Sangwoo Nam et al. The powder feeding and mixtures analyzed and optimized through regression analysis. It is understood that instead of directly building the SS316L material on the mild steel, functionally graded SS316L with “Fe” resulted in avoiding the cracks and pores at the interface in the initial layers. Also, the builds were resulted in better quality and sound in terms of no-lack of fusion, porosity, and smoother. Therefore, the printing of functionally graded materials through DED might result in better quality, however in specific to the materials involved. Further research in this newly emerging area may be required for better understanding [9].

4.5.2 Ti alloys

Ti alloys are mostly used in the aerospace and biomedical applications. Process parameters, thermal history, heat transfer kinetics, and solidification behavior greatly influence the microstructure and mechanical properties of these alloys manufactured by DED. For a better quality of build the laser input energy, powder quality, flowability, layer thickness, scan speed, and scan strategy play a vital role. Solidification mechanism and growth kinetics are also very important to obtain final microstructure and therefore the mechanical properties. The deposition rate is also important; for example, lower rates of deposition promote columnar grains whereas higher deposition rates promote fine and equiaxed grains. The mechanical properties are anisotropic with respect to the tensile axis of the build; however, it can be minimized by proper heat treatment technique. Usually, the ductility is lower and higher the strength for Ti alloys manufactured by DED compared to the conventionally processed ones [10].

The thermal history and cycling in the DED process results in the acicular α' martensitic phase in the microstructure. The acicular α' martensitic microstructure contributes the strength in DED processed Ti6Al4V samples and the mechanical properties are in comparison to conventionally manufactured samples and moreover it is observed that the fatigue limit is also higher for DED processed samples when compared to forged or wrought Ti6Al4V samples [11].

Build orientation of DED parts may have an impact on their thermal history, microstructure, and mechanical behavior. Still significant investigation required to understand how the build orientation on the tensile and fatigue behavior impacts. Plastic deformation is the key factor in fatigue behavior as the fatigue crack propagation begins by localized plastic stress and therefore the strain-controlled cyclic tests are important in determination of fatigue behavior of a material. Still no enough work has been done to understand the cyclic strain behavior of DED-manufactured components, which is very crucial for application point of view.

In situ data monitoring devices give us the better control over the DED processes and improved functionality as well as the final part quality. Furthermore, the data generated by the monitoring devices can be used for improving the process optimization/control and successful component manufacturing/remanufacturing.

Recent research suggests, however, that the melt pool area alone may not be an adequate indication of part quality. Layer height is one of the important variables apart from other process variables. Due to the rapid cooling rates experienced in DED, therefore the finer microstructure, and thus the mechanical properties of DED parts, such as tensile properties, hardness, impact resistance, and so on, are in comparable or better to those of traditionally manufactured materials. Pores and inclusions, which are intrinsic results of improperly optimized DED processes, they may be deleterious to mechanical properties [12] Figures 4.3 and 4.4 shows the microstructure and fatigue properties of direct laser deposited and LENS processed Ti-6Al-4V alloy.

4.5.3 Ni base alloys

IN 718 alloys or Ni base super alloys mostly are used for high temperature gas turbine applications due to their excellent high temperature oxidation and creep resistance.

The study on IN 718 powder material catchment behavior by using high-speed camera understands the Marangoni flow, which is depending on the materials chemistry and temperature gradients causes the powder particles to migrate toward the center of laser beam interaction in laser based DED technique. Furthermore, better geometrical tolerances in the build can be possible by the aligning the powder stream vertex to the center of the laser beam.

Temperature gradient (G) and growth rate (R) are the key factors that influence the microstructure features during the DED. Dendritic solidification continues in

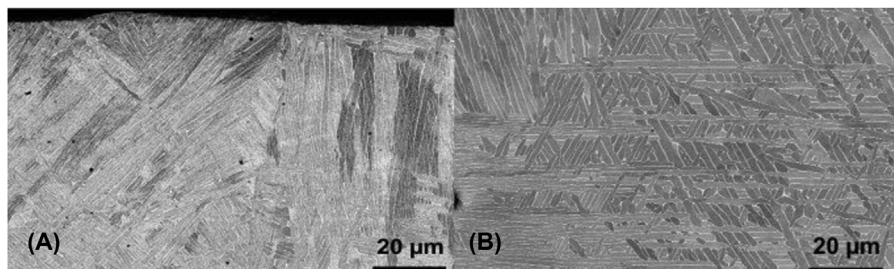


Figure 4.3 Microstructure of a direct laser-deposited Ti-6Al-4V part in Y–Z plane at (A) the top region and (B) the bottom region.

Source: From N. Shamsaei, A. Yadollahi, L. Bian, S. M. Thompsona, An overview of direct laser deposition for additive manufacturing; Part II: Mechanical behavior, process parameter optimization and control, *Addit. Manufact.* 8 (2015) 12–35.

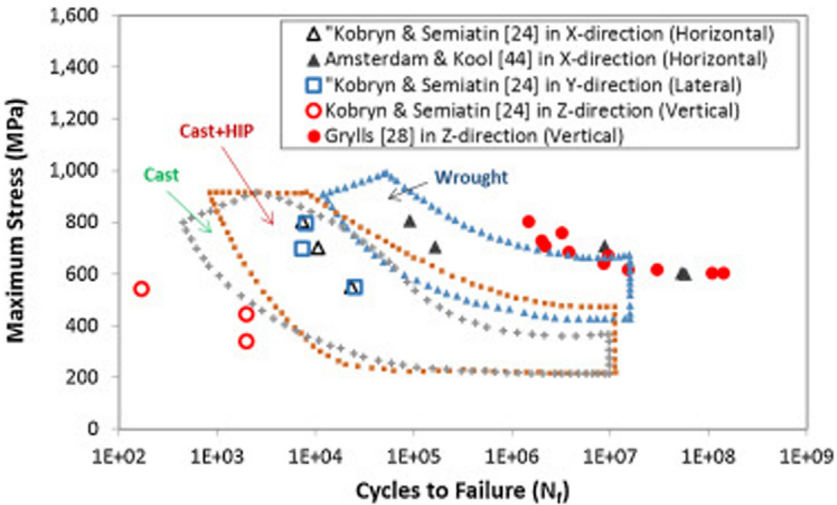


Figure 4.4 Comparison of S–N data of different studies for LENS Ti-6Al-4V specimens in different directions [13–16] to the cast, cast plus HIP, and wrought materials.

Source: From N. Shamsaei, A. Yadollahi, L. Bian, S. M. Thompsona, An overview of direct laser deposition for additive manufacturing; Part II: Mechanical behavior, process parameter optimization and control, *Addit. Manufact.* 8 (2015) 12–35.

Alloy 718 under high temperature gradient circumstances due to the presence of refractory elements [17].

Most of the earlier studies show that grain growth begins epitaxially from the substrate. A low heat input can be attained by low power or high scanning speed and it will be appropriate for creating equiaxed grains [18]. The heat input can be adjusted according to the alloy specification depending on the application [19]. Higher heat input results in a columnar microstructure with directional solidification texture of grains, whereas a lower heat input provides bimodal equiaxed and columnar microstructure with reduced grain size [20].

The single-track DED high deposition rate experiments of IN718 and IN625 (which are of comparable chemical composition) alloys reveal that IN 718 has higher porosity than IN625. It is also understood that the finer microstructure is with IN625 in comparison to IN 718. The stronger convection in IN 625 comparable to IN718 might be the reason and furthermore, the solidification rate and heat transfer kinetics are faster in case of IN625 [21].

IN-718 built through DED exhibits similar or comparable mechanical properties to the as-cast specimens. However, the DED specimens deposited at 300 W laser power are better in terms of less microstructural defects such as porosity, inclusions, etc. in comparison to the specimens deposited at 400 W [22] Figure 4.5 indicates the laser deposited IN-718 specimens mechanical properties Vs cast IN-718 specimens. Table 4.1 shows the mechanical properties different laser processed materials in comparison to the wrought counter parts.

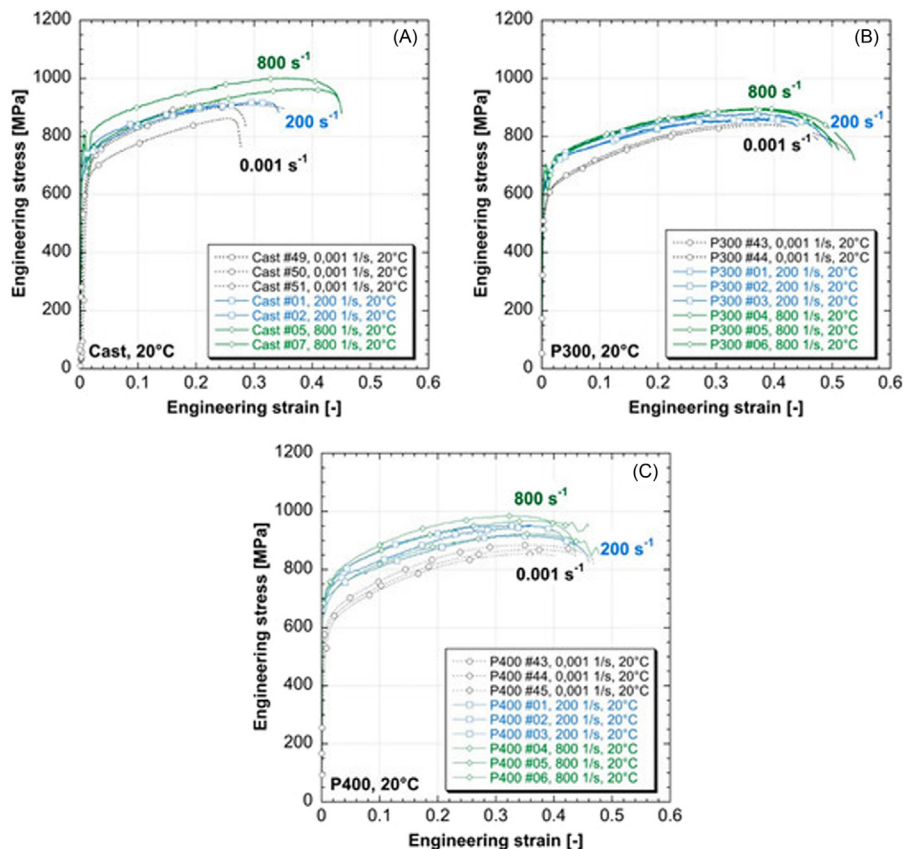


Figure 4.5 Mechanical behavior of the realized as-built Inconel 718 Specimens at different strain rates: (A) as-cast; (B) as-built at 300 W; (C) as-built at 400 W.

Source: From F. Mazzucato, D. Forni, A. Valente, E.C. Cadoni, Laser metal deposition of inconel 718 alloy and as-built mechanical properties compared to casting, Materials 14 (2021) 437.

4.5.4 Al-alloys

4.5.4.1 Direct energy deposition process parameters and their influence on the functionality of the parts

DED technologies have enormous promise for exploiting low-density material systems like aluminum (Al) and its alloys. Al and its alloys treated by DED are widely used in engineering applications such as aerospace, automotive, and consumer applications. The DED process parameters, on the other hand, have a substantial impact on the quality and functional performance of the end-use parts. As a result, the DED process parameters should be tuned in order to produce defect-free, high-density products with little distortion and residual stresses. As a result, the actual

Table 4.1 Comparison of the tensile properties of materials manufactured by direct laser deposition (DLD) and their counterparts at room temperature.

Alloys	Ultimate stress (MPa)		Yield stress (Mpa)		Elongation to failure (%)		Additive manufacturing process
	Wrought	DLD	Wrought	DLD	Wrought	DLD	
316 SS	586	758	234	434	50	46	LENS
316L SS	480 ^a	540–560	170 ^a	330–345	40 ^a	35–43	Laser consolidation
404L SS	—	655	276	324	55	70	LENS
AISI H-13	1725	1703	1448	1462	12	1–3	LENS
CPM-9V	—	1315	—	521	—	> 2	Laser consolidation
Ti-6Al-4V	931 ^b	896–1000 ^b	855 ^b	827–965 ^b	10 ^b	1–16 ^b	LENS
TC-18	1157 ^b	1147–1188 ^b	1119 ^b	1095 ^b	14 ^b	4.5–5.75 ^b	Laser melting deposition
IN-718 ^c	1379 ^c	1400 ^c	1158 ^c	1117 ^c	20 ^c	16 ^c	LENS
IN-625	834	931	400	614	37	38	LENS
IN-600	660 ^b	731	285 ^b	427	45 ^b	40	LENS
IN-690	725	665	348	450	41	49	DLF
IN-738	1095	1200	950	870	6.5	18	Laser consolidation

^aHot finished—annealed.

^bAnnealed.

^cSolution treated and annealed.

component shape will be as close as possible to the end-use application requirement in terms of geometry and functionality.

For successfully deploying DED technology to produce 3D goods with complicated geometries, optimizing DED process parameters is crucial. As a result, it is critical to better understand how the laser power and scanning speed of the DED process affect the deposited layers and their microstructural and mechanical properties of Al and its alloys [23]. The layer interface bonding qualities between the deposited layer and previously deposited layer/substrate, as well as the microstructural characteristics of the deposited layer, have been well described. Furthermore, uneven and random powder morphology influences the powder flowability, resulting in undermelting of the powder and the development of porosity in the deposited layer. Furthermore, greater feeding powder rates lead to undermelting of feeding powder, making good metallurgical bonding between the deposited layer and previous deposited layer/substrate is difficult [24]. As a result, the best powder feeding rate for Al and related alloys should be determined [24].

Riveiro et al. used a systematic factorial approach to investigate the effects of DED process parameters on the microstructures and morphology of deposited layers. By raising the powder flow rate and scanning speed, the melt morphology, such as depth and the accompanying dilution impact, is minimized. The analysis discovered that enhancing powder deposition efficacy is critical for lowering manufacturing costs [23–25]. Figure 4.6 indicates the influence of laser power and scanning speed on the DED deposited layer properties of Aluminium and its alloys.

4.5.4.2 Microstructure and mechanical properties improvement in Al alloy parts

The DED-processed parts must withstand the demands of service loading and operating conditions such as high/low temperatures, corrosive media, and so on. As a result, the performance of DED parts should be assessed in terms of their mechanical qualities. Furthermore, suitable secondary surface modification procedures could be used to improve the mechanical properties of DED parts. Deep surface rolling (DSR) is one approach that has proved to improve the fatigue resistance of DED parts.

Improvement of fatigue behavior of direct energy deposition parts

By causing nonplastic deformation events, the DED process can create compressive residual stresses on the surface of DED processed parts. Under cyclic loading, these favorable compressive residual stresses may improve fatigue crack propagation resistance. Furthermore, the greater the presence of compressive residual stress, the greater the DED parts' fatigue resistance. When compared to other techniques such as shot peening, the DSR is known to create compressive residual stresses to a depth of around 1 mm from the surface of DED parts with little surface roughness. Researchers looked at the effects of DSR and DSR + postheat treatment techniques on DED produced parts to see if they may increase the fatigue resistance of the Al

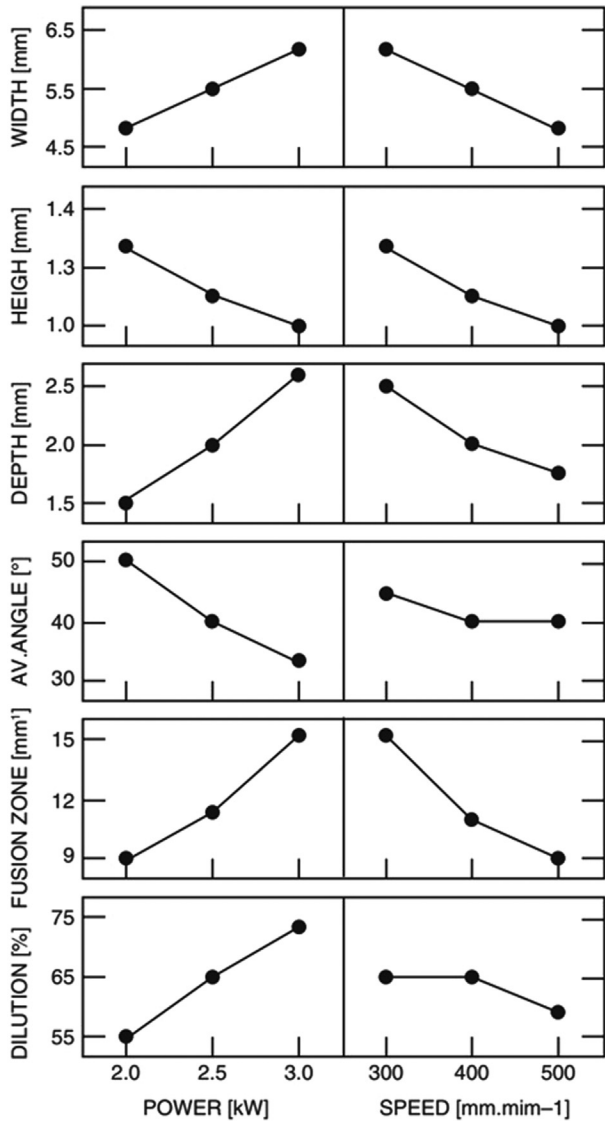


Figure 4.6 Influence of laser power and scanning speed on the DED deposited layer properties.

Source: From F. Caiazzo, V. Alferi, P. Argenio, et al., Additive manufacturing by means of laser aided directed metal deposition of 2024 aluminium powder: investigation and optimization, Adv. Mech. Eng. 9 (2017) 1–12.

7075-T651 alloy. The results showed that as compared to DSR alone, the DSR + postheat therapy approach improved fatigue life significantly. Furthermore, the DED + DSR-treated components’ beneficial influence on roughness-induced

fracture closing phenomena played a vital role in enhancing fatigue lifetimes. However, there are few research on the implications of microstructural changes such as texture development and anisotropy on fatigue performance.

Enhancement in the corrosion resistance of direct energy deposition parts

In the DED process, the desirable microstructure control depends on the process parameters and primarily on the composition of powders, such as homogeneous or heterogeneous. Subsequently, the microstructure will dictate the corrosion resistance of DED parts.

The melting temperature of Al-Si alloys is similar to that of magnesium alloys; therefore investigations into the fabrication of Al-Si layers using the DED technique resulted in a microstructure with refined grains, greater mechanical performance, and increased corrosion resistance. The Al-Si deposited layer with Al/Si powder on magnesium alloy parts (dissimilar material fabrication route) with optimized DED process parameters has been reported to improve the quality of DED parts produced with minimal porosity, resulting in superior hardness, better wear, and enhanced corrosion resistance [13,16,26–28].

Current challenges in direct energy deposition of Al alloys

Regardless of the widespread use of DED technology in practical applications, there is a need for more research and development to fully explore DED for strategic and high-end-use parts in the industrial environment for reliable and superior performance. As a result, scientists are overburdened with the task of resolving these knowledge-bridging tasks as well as the technological issues associated with DED technology.

The physics of the complicated DED process involving thermomechanical aspects responsible for the high quality of the end-use DED products is used to address the technological obstacles. Intricate relationships between processing, microstructure, mechanical, and functional properties and their characteristics of DED parts and microstructural behavior defect development solidification behavior and grain shape and morphology are all important considerations that must be addressed right away.

In addition, progress toward a full deployment of DED-based AM in the industrial setting has been hampered due to presence of technological concerns and obstacles such as qualifications, certification, geometric constraints and complexities, scale-up of construction strategies, process design and printing sequence, lack of standards, postprocessing procedures, and safety and health. Therefore these area required research attention. Economic feasibility difficulties such as cost competitiveness, low market share, feedstock material compatibility, intellectual property and cybersecurity, and a lack of practical and focused direction for DED processing are all important roadblocks to DED technological advancement. It is worth noting that the concerns and challenges stated above are interconnected, as is the impact of

them, notably on the deployment of metal DED. As a result, these issues can be addressed by accelerating research and developing a better grasp of DED technology requirements and policies in order to enter the relevant application market [14,29–35].

4.5.4.3 Future scope for direct energy deposition of Al alloys

Postprocessing technique for improving the quality of direct energy deposition parts

With additional modification and employment of allied techniques concurrently, or postprocessing techniques with the DED processing set up, DED-treated end-use parts are projected to outperform in their respective applications. Recently, laser posttreatment on cold spray Al alloy coatings has been investigated. The beneficial effects of laser as a posttreatment have resulted in reduced surface porosities and thus improved corrosion resistance and facilitate the formation of the intermetallic phase Ni-Al system, and defects such as pores, cracks, and inhomogeneity can be significantly reduced. Similar beneficial effects can be envisaged by employing laser posttreatment on the DED processed Al alloy parts.

Development of direct energy deposition amenable unconventional class of material system

Only a few grades of Al alloys are appropriate for DED processing at the moment; as a result, there is a scarcity and a need to speed up the research and development operations. The intrinsic thermal features of DED processes generate unconventional microstructures and mechanical properties, so development and research must focus on the essential phenomenological aspects of the DED process, such as directional heat extraction, rapid solidification, and repeated melting and reheating or intrinsic heat treatment. As a result of addressing these issues, the number of DED-enabled material alloy systems will increase, and the DED technology's full potential will be realized. Furthermore, recent research suggests that including nanoparticles into the feedstock can enhance the printability and quality of the final end-use item.

Induction of grain refinement mechanism in direct energy deposition process

It is discovered that an alternating electric field can produce equiaxed microstructure rather than dendritic architecture. The electric field has been shown to improve nucleation rate and grain refinement in the literature. The nucleation rate can be improved and grain refinement achieved while the current density is in a specified critical region. The skin effect of alternating current tends to limit alternating current to the surface of the conductive melt, and because the solid resistivity is lower than the melt, current preferentially travels through the solid phase. Because of the low temperature of the substrate, the melt pool solidifies first toward the bottom, where the current concentrates, resulting in a higher current density at the bottom

of the deposited layer. Increased grain refinement is linked to higher current densities, which causes refinement in grains at the top and bottom of the deposited layer. The external environment has an impact on grain refinement in the top section, resulting in a faster cooling rate. However, grain coarsening occurs in the middle of the deposited layer due to insufficient current density for grain refining, Joule heating, and relatively slow cooling rates investigated the effect of molten pool stress distribution in static and dynamic electromagnetic fields. When compared to static fields, rotating fields were found to have a more robust ability to stabilize the stress field. In laser cladding, the aforementioned works show how to modify/control grain size and distribution of alloying elements, improve microhardness and corrosion resistance, and reduce cracks and dilution [15,36–45].

4.6 Conclusions

The current chapter presents the overview of DED manufacturing process, its advantages and disadvantages, and applications. Furthermore, it details about the microstructure and mechanical properties of different metallic materials such that Al alloys, steels, Ti alloys, and Ni -alloys manufactured through DED. Overall, the powder characteristics, thermal cycling/history, process parameters, heat transfer kinetics, solidification behavior such as the solidification rate, thermal gradient, and heat treatment greatly influence the part quality and functional performance of the DED-processed materials. The build quality and performance can be greatly improved by controlling or optimizing aforementioned key parameters during the component manufacturing/remanufacturing by DED.

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Postprocessing challenges in metal AM: Strategies for achieving homogeneous microstructure in Ni-based superalloys

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5.1 Introduction

Manufacturing is the process of converting the raw material into processed component suitable for end use. The manufacturing techniques are classified into three categories namely subtractive, additive, and hybrid manufacturing. As their name suggests, subtractive manufacturing involves removal of material as in machining, turning, etc. whereas additive processes involve addition of material as in welding, 3D printing etc. Hybrid techniques involve processes such as casting and injection moulding. Additive method is considered a better approach than subtractive manufacturing because it involves lower wastage of raw materials, low stress introduction, and ability to produce complex shapes [1].

Additive manufacturing (AM) or 3D printing is an approach in which the component is manufactured in a layer-by-layer manner. In this process, a 3D CAD model of the component to be manufactured is divided into thin slices (i.e., slicing) and this data is fed into the 3D printer. The printer then deposits the raw material in a layer-by-layer manner as per the input in slicing step [2–5]. In the recent years, the process has changed substantially due to the developments, which has allowed a variety of materials that can be processed by AM. AM in its true form still has capabilities that needs further investigation and is probably the reason that tech giants like NASA, SpaceX, ISRO, etc. are looking toward AM in their future missions. NASA in the recent “Centennial Challenge” had asked to create sustainable housing solutions for “Earth and beyond” using 3D printing for deep space explorations.

In its early years, the process was termed as ‘rapid prototyping’ technique as it was used to produce visualization models and prototypes from polymer based materials. With the advent of technology and processing capabilities, the process can now also be employed on a variety of materials such as biomaterials, biopolymer composites, metals, and concrete. Due to this flexibility in the materials that can be

processed with AM, the process has been widely employed in aerospace, automobile, medical and dental, and defence applications [2,6].

The AM techniques are classified into different categories depending upon the physical state of raw material such as extrusion based, powder based, and liquid based as well as the manner in which the material is fused such as Fused deposition modelling, direct metal laser sintering, stereolithography, direct light processing, etc. Metal AM processes are distinguished depending on the heat source employed (laser beam, electron beam or electric arc), feedstock material processed (powder or wire), and the feed system. Based on these factors, AM processes are categorized as powder bed fusion (PBF), directed energy deposition (DED), and binder jetting (BJ) [2–6]. PBF is a technique in which the metal powder is heated/melted using a laser beam or electron beam. DED is a technique in which the feed material (powder or wire) is fed through a nozzle and is melted using laser/electron beam or electric arc and deposited layer by layer. BJ involves a print-head selectively depositing a liquid binding agent onto a thin layer of powder particles followed by sintering [7]. DED and PBF are most widely used processes because of the flexibility offered by the techniques and the ability to fabricate intricate shapes and geometries. The basic principle of operation of these processes is discussed below.

5.2 Direct energy deposition

DED is a metal AM technique involving the use of thermal energy to fuse metal (either powder or wire) by melting while deposition as shown in Fig. 5.1. DED

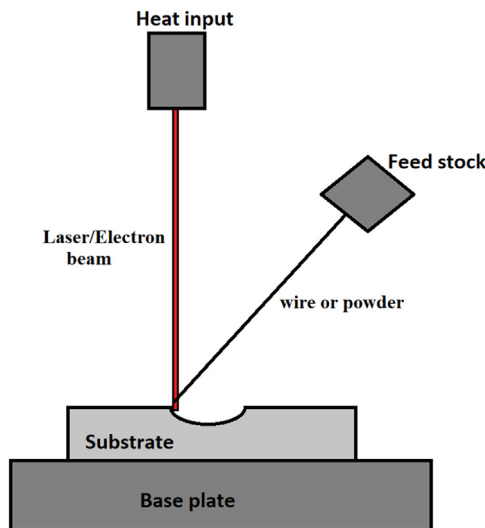


Figure 5.1 Schematic showing directed energy deposition (DED) process during repair applications.

process allows the fabrication of parts by melting metal (powder or wire) as it is being deposited [8]. A laser source, electron beam or plasma arc can be used as the heat source. This method is also known as laser engineered net shaping (LENSTM) and laser cladding. As the heat source is moved over the substrate, the energy is focused on a small area of the component while the feedstock is melted and the molten metal is deposited. As the heat source moves away, the deposited metal solidifies [9].

The technique can be utilized in repair applications such as filling cracks for high-value aerospace components. It can be used for repairing components such as compressors, turbine blades and guide vanes, combustion chambers, and blade discs. The system can also be employed for depositing different materials and can fabricate a functionally graded component [9,10]. The mechanical properties of the AM manufactured component depend upon process parameters such as laser power, scan speed, feed rate, etc. [11].

5.3 Powder bed fusion

PBF technique employs a power source (laser or electron beam) to scan and melt a layer of fine metal powder to build up a component in a layer-by-layer manner as shown in Fig. 5.2. After a layer is deposited, fresh metal powder is spread over the entire surface using a recoater and the power source melts the fresh powder layer, and a new layer is deposited. PBF provides the ability to produce in-process support structures for overhangs and undercuts [11]. Thus, highly complex shapes with high geometric accuracy can be manufactured. PBF has an edge over DED technique in producing complex geometries [10].

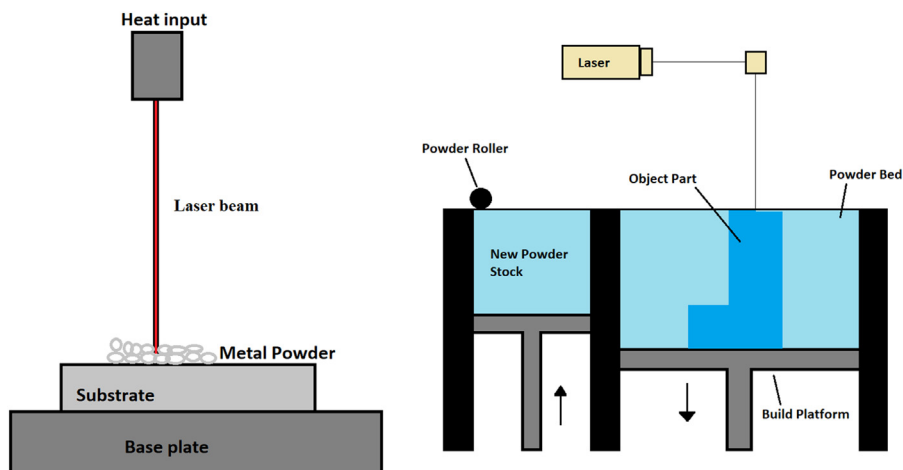


Figure 5.2 Schematic showing the powder bed fusion process (PBF).

PBF because of its higher resolution has the capability to create internal passages in high-value components such as gas turbine blades. The resolution of the PBF process depends on the size of metal powder used, scan speed, laser parameters, and heat transfer to the bed [12]. For repair applications, PBF in the standard form is difficult to perform because of the mechanism employed by the printer. The recoater's movement would be hindered by the solid (already deposited) part and could lead to problems such as damage to machine and lack of fusion. Therefore, certain additional attachments are required to carry out the repair procedure [13].

PBF is widely used for manufacturing complex geometries because of high-dimensional accuracy and capability for topology optimization. PBF has the capability to create internal passages in high-value components such as gas turbine blades which is not possible with other metal AM techniques. Therefore, PBF is chosen over other AM processes because of its ability to manufacture intricate geometries.

Inconel 718 is a high strength superalloy of nickel and exhibits thermal stability at elevated temperatures around 600°C and has excellent resistance to wear, oxidation, creep resistance, and superior fatigue life. IN718 has been used as the main structural material for aircraft parts such as turbine blades, guide vanes, engine manifold, and combustion chamber. To fabricate parts with complex geometries and merging components to reduce the number of parts, AM presents a feasible approach (Fig. 5.3).

Fig. 5.3 shows the transformation-time-temperature (TTT) diagram for Inconel 718 alloy system. ($\text{Ni}_3(\text{Al}, \text{Ti})$) and (Ni_3Nb) are the primary strengthening phases for IN718. Apart from these, IN718 has other phases such as phase (Ni_3Nb), Laves phase ($\text{Fe}, \text{Cr}, \text{Ni}, \text{Si}$)₂ (Nb, Mo), and carbides (M_{23}C_6). These phases are termed as detrimental phases as they deteriorate the mechanical properties and reduce the fatigue and creep resistance of the component. In AM, the composition of phases formed varies from cast components because of rapid heating and cooling. The microstructure and composition of the phases in the as-printed component are dependent on the process parameters such as laser power, scan speed, scan strategy, hatch spacing etc. Columnar microstructure is preferred with strengthening phases and for high-temperature application.

5.4 Crystal growth theory

Crystallization is the process through which the atoms, molecules, or ions arrange themselves in a repeating pattern. The process is divided into two stages namely nucleation and growth. In the nucleation stage, nuclei are formed in the melt pool. The number of nucleation events per unit volume per second is known as the nucleation rate. In the initial stage, the nucleus formed is dissolved back into the melt pool if not oriented correctly. Once the nucleus is stabilized, the growth rate ensues and the crystalline structure propagates outward from the nucleating site. The

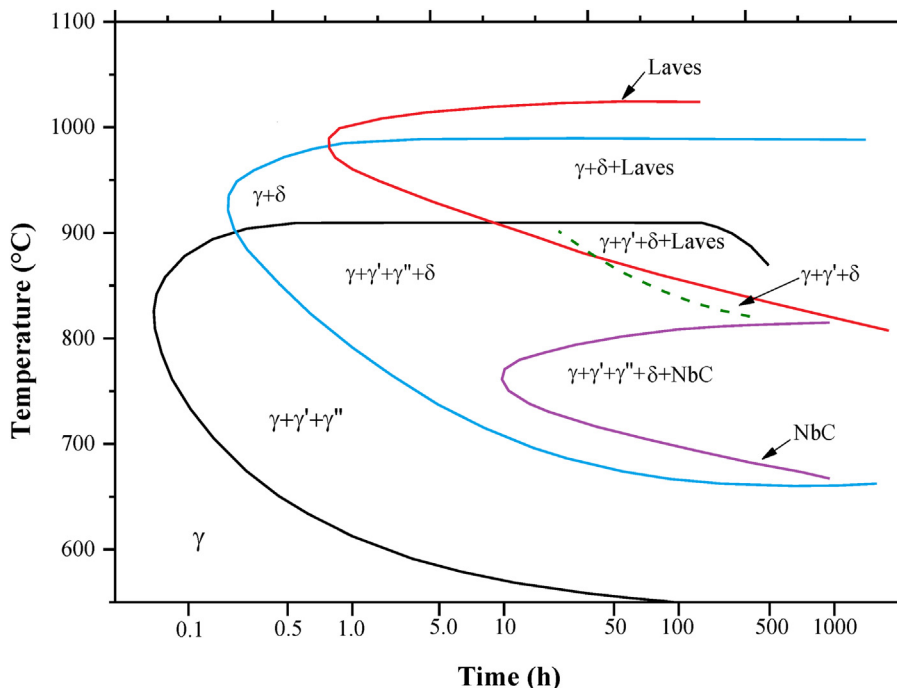


Figure 5.3 Transformation-time-temperature diagram of Inconel 718 alloy system.

growth of nucleus depends upon on the presence of low energy sites such as steps. Rate of increase of the size of the growing particle is termed as growth rate.

Within a melt pool, atoms arriving from the liquid onto crystal surface will have a higher probability of remaining on the crystal if they diffuse at steps in the surface than if they arrive at atomically smooth surface. It follows from this, that growth of the crystal will take place by an edgewise extension of the more closely packed planes rather than by growth normal to any particular lattice plane. Growth normal to closely packed planes will require either the existence of defects or the nucleation of an extra plane of atoms for which the activation energy will be relatively high.

5.5 Grain morphology control

The microstructure evolution is mainly determined by the ratio of temperature gradient (G) to the solidification rate (R). The temperature gradient acts as the driving force for the grains to grow and determines whether growth rate or the nucleation rate will dominate. Solidification rate is the rate at which the heat is lost from the metal molten pool thereby determining the rate at which the pool will solidify.

In laser PBF (LPBF), by the nature of the process, we achieve columnar grains for alloy systems. This is because the small scale localized solidification events in LPBF process occur under steep temperature gradients followed by high cooling rates favouring the formation of dendritic-columnar structures during solidification [14]. It is observed that G/R ratio near the bottom of the melt pool fall in the region for columnar-dendritic solidification, whereas the equiaxed solidification condition may be satisfied near the melt pool surface where the thermal gradient is lower than at the bottom of the pool [15]. Typically, the G/R ratio produced by LPBF process is far below the lower limit for planar solidification to occur. Therefore, in order to achieve the desired crystallographic features, there is a need to control the processing parameters to fulfil the necessary conditions for obtaining the required crystallographic features (Fig. 5.4).

For obtaining the required texture, G/R ratio and preferential growth directions in the crystal have to be taken care of. In the processing of the component, certain cases can be encountered with respect to different values of G and R , which are as discussed below. These cases have been discussed from the basic understanding of the effect of temperature gradient (G) and solidification rate (R) on the microstructure (Case 5.1) and the resultant microstructure are shown in Fig. 5.5, 5.6, 5.7 and 5.8.

Case 5.1: Low G , high R .

Since the temperature gradient is less, that is, the temperature difference between the molten metal and solid underlying layer is less, grain growth will dominate. But since the solidification rate is also high, the temperature gradient suitable for growth rate to dominate cannot be maintained for long and soon nucleation rate will dominate. Due to Marangoni convection, heat is also lost through sides of the melt pool along with loss through conduction in the solid layers as shown in Fig. 5.9.

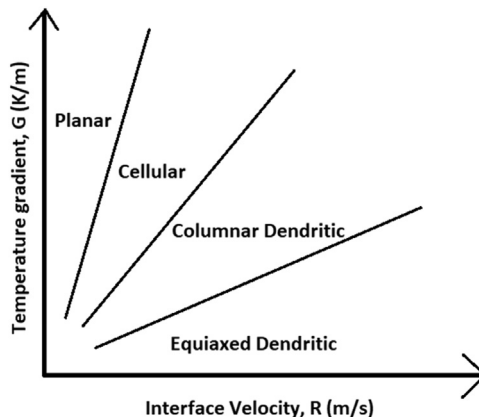


Figure 5.4 Schematic representation of the combined effect of thermal gradient and interface velocity on solidification microstructure.

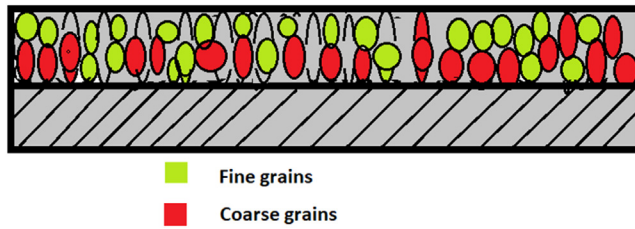


Figure 5.5 Microstructure showing mixed, i.e., coarse and fine grains for Case 5.1.

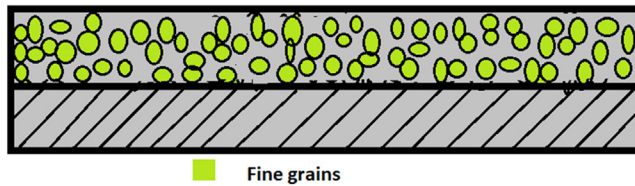


Figure 5.6 Microstructure showing fine grains for Case 5.2.

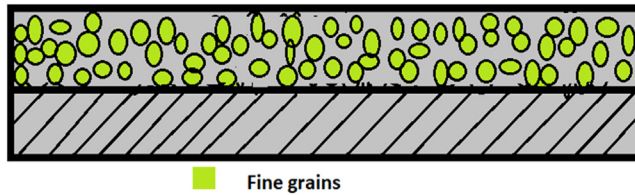


Figure 5.7 Microstructure showing fine grains for Case 5.3.

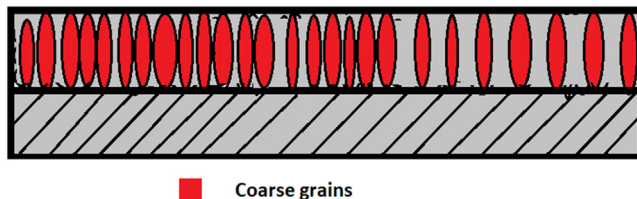


Figure 5.8 Microstructure showing coarse grains for case 5.4.

Case 5.2: High G , high R .

Since the temperature gradient is high, nucleation rate will dominate, which is further enhanced by high solidification rate. This leads to the formation of finer grains.

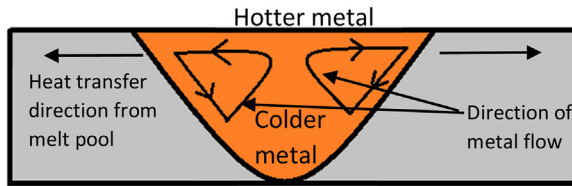


Figure 5.9 Schematic showing transfer of heat through Marangoni convection through flow of molten metal.

Case 5.3: High G , low R .

The temperature gradient is high and therefore nucleation rate will dominate leading to the formation of fine grains. Low solidification rate will not be affecting the formation of coarser grains as there isn't enough driving force to lead to the formation of coarse grains or growth rate to dominate. Thus fine grains are obtained.

Case 5.4: Low G , low R .

In this case, growth rate will dominate. Since solidification rate is low, the temperature gradient will be maintained for longer duration. The effect of Marangoni convection is not sufficient to increase the temperature gradient as conduction is dominant mode of heat loss. So temperature gradient is maintained. This leads to enhanced grain growth and the formation of coarse grains.

5.6 Hotter metal

Columnar grains are formed in selective laser melting due to the direction of heat transfer. The grains grow toward the molten pool along the z -direction, since the growth velocity is much higher when the crystals growth direction is aligned with the maximum temperature gradient. Microstructure evolution is mainly determined by the ratio of temperature gradient (G) to solidification rate (R). At the bottom of the melt pool, G has the largest value due to decreasing heat input and R is also very low. Therefore G/R is large.

Since the heat is extracted from melt pool to the previously consolidated layers and the substrate. In case of pure metals, this leads to stabilization of solidification front (i.e., planar solidification mode). For pure metals, solute redistribution is not relevant. When a liquid phase of crystalline metal and a substrate are at the same temperature and the liquid wets the substrate, the effective contact angle approaches 0 degree, and critical nucleus size approaches atomic dimensions. Thus, solid growth into the liquid requires only that individual atoms in the liquid surrenders their latent heat and join the existing crystal structure of the adjacent solid. This is epitaxial growth due to planar solidification.

For alloys, due to solute redistribution, the liquid may become undercooled. This leads to destabilization of solidification front and a transition from planar to dendritic or cellular solidification mode. Thus, dendritic growth will take place when the supercooling is large.

For pure metals, this transition in solidification mode occurs when the thermal gradient in the liquid phase at the solidification front becomes lower than the critical gradient. This transition in the solidification mode depends on the thermal gradient in the liquid and the critical thermal gradient as shown in Eq. 5.1.

$$\frac{\delta T}{\delta x_{crit}} = - \frac{C_0 (1 - k)}{\frac{D_L}{R} k} \frac{\delta T_L}{\delta C}, \quad (5.1)$$

where $\frac{\delta T}{\delta x_{crit}}$ is critical thermal gradient, C_0 is solute concentration, k is solute partition coefficient, D_L is solute diffusion coefficient in liquid phase, R is solidification growth rate, $\frac{\delta T_L}{\delta C}$ is gradient of equilibrium melting point.

From Fig. 5.10, we can see some grains aligned in the horizontal direction at the top of the melt pass. The columnar grains grow toward maximum temperature gradient in the melt pool, which is concave in shape. Grains grow from the boundary toward the centre of the melt pool. Therefore the grains that start to grow toward the top of the pool grow in the horizontal direction. However, the top of the deposited layer is remolten during the application of the subsequent layer. This leads to remelting of all grains near the top of the previously solidified melt tracks as shown in Fig. 5.8. This is known as smearing effect. Due to this, the horizontally aligned columnar grains are remelted and upon resolidification they align themselves along the z -direction in the direction of maximum temperature gradient [16].

The effective volume of the molten pool and the volumetric laser energy required per unit volume can be estimated from Eqs. 5.2, 5.3 and 5.4 [17].

$$V_m = \frac{1}{2} \times \frac{4\pi R^3}{3} - \frac{\pi(R - H)(R^2 + r^2 + Rr)}{3}, \quad (5.2)$$

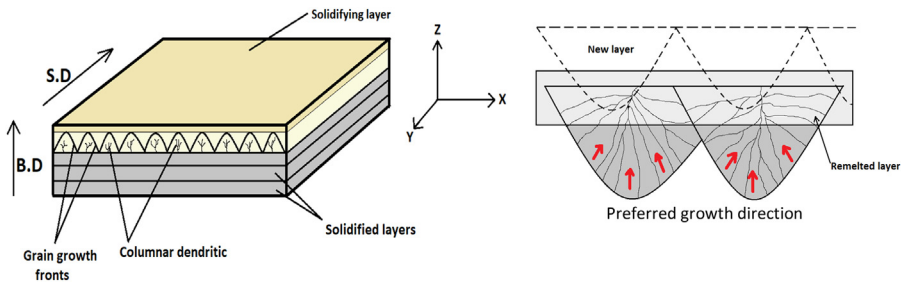


Figure 5.10 Schematic showing the smearing effect.

$$Q_v = \frac{\pi r_b^2 E_{ef} \Delta t}{V_m}, \quad (5.3)$$

$$E_{ef} = \frac{2\alpha P(1 - e^{-2})}{\pi r_b^2} - E_{loss}, \quad (5.4)$$

where r is effective radius of laser beam or melt zone radius, H is the effective laser melting depth, r_b is laser beam spot size, R is the radius of the sphere, Q_v is the volumetric laser energy per unit volume, E_{ef} is effective laser energy flux, t is laser exposure time, α is absorptivity coefficient, P is laser power, and E_{loss} is the energy loss.

The presence of horizontal grains in the melt pool in the intermediate layers of the component can be altered by the deposition of the subsequent layer but when the final layer is laid off, these grains are left on the top. The horizontally aligned grains in the top layer can be altered by scanning the laser on the top surface remelting a small volume of the top layer. This will lead to reduction in the volume of number of grains aligned horizontally.

When crystalline solids grow into a supercooled melt, growth occurs in the characteristic crystallographic directions called dendritic growth directions, preferred growth, or easy growth directions. For different crystal structures, these growth directions can be different. For body centred cubic (BCC) it is $\langle 100 \rangle$, for face centred cubic (FCC) it is $\langle 100 \rangle$ and for hexagonal close packed structure it is $\langle 10\bar{1}0 \rangle$.

If one considers both the positive and negative orientations of these three preferred growth directions, each of these three crystallographic forms has six possible directions. At any instant, the operative growth direction will be along which the steepest temperature gradient exists. This means that crystal will grow preferentially in $\langle 100 \rangle$ direction because it is oriented preferentially in the building direction along which the maximum temperature gradient exists. For BCC or FCC crystal $\langle 100 \rangle$ being the easy growth direction, they grow quickest when $\langle 100 \rangle$ crystal direction is aligned with the maximum temperature gradient.

There is a predominant $\langle 100 \rangle$ easy growth direction in the plane of the sheet and a $\langle 110 \rangle$ difficult growth direction at right angle to $\langle 110 \rangle$. Partial remelting of underlying layers removes horizontally grown grains at the top of the melt pools. This results in the smearing effect of the $\langle 100 \rangle$ texture.

5.7 Effect of additive manufacturing processing parameters on metallurgy

5.7.1 Laser parameters

The LPBF process parameters include laser power (P), scanning speed (v), point distance (PD), exposure time (θ), hatch spacing (h), stripe width (w), stripe overlap

(γ), beam offset (δ), and layer thickness (t). These parameters should be controlled during the fabrication process to control the mechanical properties.

Laser power first hand controls the energy supplied to the powder layer. It affects melt pool shape and size, grain size, texture and the thermal residual stress profile. Scan speed/rate is the speed at which the laser beam scans over the powder layer, melting the metal powder. It determines the interaction time of the laser beam with the metal powder, which in turn determines the energy provided to the powder. With an increase in scan speed, the interaction time decreases along with the energy supplied to the metal powder.

AM is a progressive layer-by-layer process where a layer is made of a number of line scans. This introduces another important parameter called hatch spacing (h), defined as the distance between two adjacent line scan tracks. In the LPBF process, the layers in components are fabricated in a track-by-track manner. The hatch spacing decides the level of overlap between two adjacent layers. This determines the heat input to the powder layer and the track morphology thus formed determines the surface quality [18].

Therefore in order to identify the influence of each process parameter on the quality of the component fabricated, energy density has to be looked at. The laser energy density, E (J/mm^3), is defined as follows:

$$E = \frac{P}{(v \times h \times t)}. \quad (5.5)$$

The energy required for melting a certain material is dependent on the thermal properties and is estimated using the following equation:

$$E_m = c\rho(T_m - T_a), \quad (5.6)$$

where c is the specific heat capacity ($\text{J}/\text{kg K}$), ρ is the material density (kg/mm^3), T_m is the melting temperature (K), T_a is the ambient temperature (K), and E_m is the melting energy density (J/mm^3).

The influence of heat source power (P) is considered in combination with the scanning speed (v) by means of linear energy density. The linear energy density has a significant effect on the size and shape of the melt pool and the microstructural aspects of the fabricated component. Empirically, it is observed that the density of the part increases with an increase in the laser energy density along with a reduction in the porosity. However, the thermal properties of the material along with the laser energy density determine the quality of the part fabricated and resultant residual stresses. Thus, the melt pool dimensions and microstructural characteristics are determined by the incident energy and the thermal properties of the metal [12,19–21].

With the increase of E , the size and the volume fraction of laves phase in interdendritic boundaries become larger and higher. Also the density of the precipitated phases of γ'' and γ' is getting small and the particle spacing is becoming large with

increasing E [22,23]. When E increases, the microsegregation of alloying elements of Nb and Mo becomes easier and thus Laves phase is more easily formed in the interdendritic boundaries, which result in the decrease of the solution contents of Nb and Mo in γ -NiCr. Therefore, lattice parameters of γ -NiCr become smaller and γ -NiCr diffraction peaks shift to larger angles [24–27].

Watring et al. [28] reported that at low energy density of 38 J/mm^3 , the energy was insufficient to fully melt the powder causing the presence of porosity which reduced with an increase in the energy density to 65 J/mm^3 . An increase in the grain size with increase in VED (volumetric energy density) from 35 to 65 J/mm^3 is observed. Fabrizia et al. [29] observed that the numbers and size of pores decreased with an increase in the VED up to a threshold of 130 J/mm^3 . Any further increase in the energy density was found detrimental, which can be ascribed to defects of layer inhomogeneities. Liu et al. [30] reported that texture effect enhanced the anisotropy with an increase in the energy density.

From the literature, it can be inferred that increasing the energy density increases the part density while maintaining the complete melting of the powder layer. Above this, very high energy density will cause the microsegregation of elements and in some cases may lead to vaporization of the elements such as chromium. Thus affecting the composition of the parts produced. The use of high energy density also introduces high thermal stresses resulting in a large amount of tensile residual stresses in the fabricated part.

5.7.2 Scan strategy

The temperature distribution in LPBF process varies rapidly with the movement of the laser beam leading to the buildup of a large temperature gradient caused by high energy input at a localized zone. This further causes high residual stresses in the final component. Scanning strategy is one of the most influential parameters in LPBF process controlling the quality of the component fabricated.

Scanning strategy is the spatial moving pattern followed by the energy beam, which affects the thermal gradient in the component. Different strategies can be opted for countering the challenges faced during AM process such as balling effect, distortion, and large residual stresses. High-temperature gradient result in high residual stresses, which affects the critical mechanical properties such as tensile strength and fatigue life. The scanning strategy is varied by varying scanning vector rotation angle, vector length, scanning directions, and hatch spacing [31]. Some of the scan strategies are as shown below in Table 5.1.

The heat accumulation in the metal powder can be controlled by selection of optimal scanning direction and scanning sequence. In order to change the temperature distribution during fabrication, scanning sequence can be changed. Different scanning patterns involve variations in the sequence of interaction with laser parameters resulting in different reheating and remelting cycles, which make cooling rate, local melt flow, and local heat treatment in the heat affected zone near the melt pool different [37]. The temperature gradient, direction of heat flow and

Table 5.1 Effect of scan strategies on the microstructural characteristics and residual stresses [31–36].

S. no.	Scan strategy	Characteristics
1.	Island scan	• Less residual stress • Less deflection • Large columnar grains • Fine elongated grains in the boundary region of pattern
2.	Line scan	• Equiaxed grain in the centre • Maximum induced residual stress • Less deflection
3.	45-degree line scan	• Bimodal grain structure • Minimum induced residual stress • Low deflection in <i>X</i> and <i>Y</i> direction
4.	45-degree rotate scan	• High residual stress • Less deflection
5.	90-degree rotate scan	• High residual stress • Less deflection
6.	67-degree rotate scan	• Directional columnar grains • Less residual stress • Low deflection
7.	In-out scanning	• High residual stress • Maximum deflection (mostly in central region)
8.	Out-in scanning	• High residual stress • High deflection/distortion
9.	Progressive scan	• Low residual stress • High deformation
10.	Helix scan	• Low deformation • Low residual stress
11.	Alternating block scan	• low overlap • Undesirable interdendritic phase due to insufficient overlapping
12.	Rotated stripe scan	• High overlap in the melt pool • Elongated grains • Fine cellular grain structure

cooling rate have significant effects on the microstructure, grain size, texture, and grain aspect ratio.

The G/R ratio during fabrication decides the grain structure and grain size in the component. Here, G is the thermal gradient in K/mm and R is the interface speed in mm/s. The G/R determines the change in the solidification modes from columnar to equiaxed. Interlayer scan vector rotation helps in changing the direction of the heat flux during the fabrication process. This helps in achieving uniform temperature distribution leading to the generation of isotropic properties.

The residual stresses in the as-printed component can be reduced by using a shorter scan vector length. It also helps in improving the mechanical properties of the AM components. Various scan strategies with small scan vector length are island scan, helix scan, and fractal scan strategy.

Based on the various studies on the effect of scanning strategies on the microstructural evolution and residual stress, Amirjan [37] reported that the part density can be increased by interlayer rotation and resulted in a uniform microstructure by remelting of the layers. Local heat treatment performed as a result of island strategy relieved residual stresses. Geiger et al. [38] revealed that scan strategy influence the anisotropy of elastic properties and the texture could be locally tailored by adjusting scanning strategies during buildup process. Papula et al. [39] stated that interlayer rotation of 66 degrees decreases the texture and isotropy in the printed components.

The cooling rate and the temperature gradient are directly affected by the scanning strategies thus affecting the mechanical properties of the fabricated parts. It was also confirmed by Koutny et al. [40] that the reduction of the temperature gradient between the layers has a significant effect on the mechanical properties. It was observed that the island strategy gave the maximum part density whereas the meander strategy gave the best mechanical properties. However, there are a number of factors involved such as:

5.7.3 Rotation of scan vectors

The scan rotation angle between the AM build layers can be adjusted to optimize the microstructure and performance of the fabricated component. Liu et al. [41] studied the influence of scan rotation angle on mechanical properties. It was observed that scan strategy *X* gave better results than *XY45*. It has been mentioned earlier that the rotation of the scan vector benefits the microstructure and mechanical properties. However, the microstructure obtained using *XY45* was responsible for inferior mechanical properties as compared to scan strategy *X*.

Li et al. [42] observed that the specimens prepared by unidirectional scanning with 90-degree rotation exhibited better tensile properties and finer microstructure as compared to unidirectional scanning. This is because there is a more uniform temperature gradient achieved with the introduction of rotation in scan vectors.

Leicht et al. [43] analysed the effect of scan rotation on microstructure and mechanical properties of the stainless steel 316 L. It was observed that 45-degree and 67-degree scan rotation have high-angle grain boundaries. It was observed that the samples produced without rotation had higher tensile strength, yielded strength and ductility but reduced hardness showing the highly anisotropic nature of the samples.

Robinson et al. [44] analysed unidirectional scan and chessboard scan strategy. It was observed that unidirectional scan vectors oriented the residual stresses in the scanning direction. The chessboard strategy significantly reduced the isotropic nature of residual stresses in the parts. *XY* strategy gave the most uniform distribution and the lowest residual stress.

Li et al. [45] investigated the effect of scanning strategy on the distortion of Ti64 bridge structure. In this study, four different scan patterns were compared and it was observed that the pattern with 90-degree rotation of scan vector produced components with least distortion. As the scanning vector rotation increases, the curling angle of the bridge decreases.

5.7.4 Length of scanning vectors

Length of scanning vectors determines the thermal energy provided to the component. Thus the mechanical properties are also affected by this. Promoppatum et al. [46] reported that scanning length has a more distant impact on the applied energy input. Reduction in scanning lengths reduced the residual stress. Under the shorter scanning vectors, the scanning time interval between two adjacent scanning vectors is reduced. Therefore, the subsequent printing is carried out at a much higher surface temperature, which helps in reducing the temperature gradient. This is the reason behind reduction in thermal stresses generated.

The size of the island fabricated is directly related to the length of the scanning vector and has a significant influence on the residual stress. Zaeh and Branner [47] also reported that the island scan strategy also fabricates parts with low levels of stress as compared to uni- and bidirectional scan strategies.

In the island scan strategy, heat dissipation is faster as each island acts as radiating unit. Thus the heat accumulation is lower and the temperature gradient is smaller as compared to unidirectional strategy [48]. The shorter scanning vector reduces the scanning time interval between adjacent scanning vectors. This helps in maintain the surface temperature higher than the powder temperature, which reduces the temperature gradient [46]. Lu et al. [49] studied the effect of island size on the mechanical properties. Island of sizes 2×2 , 3×3 , 5×5 , and 7×7 mm² were fabricated. The island size of 2×2 mm² exhibited lowest residual stress when compared to other sizes. However, distant cracking was observed in the 2×2 specimen, which might have released a large portion of the stress. Therefore, 5×5 specimen was optimal candidate due to reduced stresses and high relative density.

Three different scan strategies were simulated by Ramos et al. [50] namely unidirectional, bidirectional, and alternating scan strategy. It was observed that the alternating scan strategy dispersed the heat throughout the part due to change of scan sequence causing more symmetric deformation. A strategy was designed in which adjacent islands are not scanned consecutively. This strategy leads to shorter melt pools, which is beneficial in reducing the residual stresses and deformation in fabricated parts.

5.8 Effect of heat treatment on metallurgy

Metal AM process involves rapid solidification of the molten metal powder layer. Because of the nature of the process, large residual stresses are introduced along

with isotropic mechanical properties. In order to make the fabricated component suitable for end application, heat treatment is used to relieve the inherent residual stresses and bring homogeneity in the microstructure. Certain heat treatment strategies are designed to dissolve the undesirable phases in the matrix leaving only the strengthening phases in the metal matrix.

For AM Inconel 718, the as-printed microstructure contains brittle laves phase formed due to microsegregation of Nb and Ti. Thus, a heat treatment strategy is required to dissolve undesirable phases back in the matrix, relieving residual stresses, and homogenize microstructure. Generally, the as-printed component is subjected to number of heat treatments namely homogenization treatment (1080°C for 1.5 hours), solution treatment (980°C for 1 hour), and double ageing (720°C and 620°C for 8 hours each). In addition to these, HIP (hot-isostatic pressing) at 1180°C for 100–150 MPa is also used to reduce the porosity level of the component [22,26].

The complex and rapid solidification and cooling during AM process inhibit effective precipitation of the strengthening phases. It is important to design a heat treatment cycle based on the properties of the as-printed component. It is required so that a certain heat treatment temperature and duration are achieved, as required to maintain a balance in the phases available in the processed component.

5.9 Solution treatment

Solution treatment involves heating of an alloy to a suitable temperature and holding it at that temperature for a suitable period of time to cause one or more constituents to enter into a solid solution and then cooling it rapidly to hold the constituents in the solution. Solution treatment for Inconel 718 alloy is carried out at temperatures above 980°C for 1 hour. Researchers have carried out the treatment at different temperatures to identify the effect on the metallurgical characteristics of the component.

The purpose of solution treatment is homogenization of Ti, Al, and Nb distribution in the matrix by dissolving the Laves and δ phases. This helps in effective precipitation of γ' and γ'' phase. Liu et al. [51] reported that with an increase in the solution treatment temperature, the δ phase disappeared at 1020°C. It was observed at 980°C, the ultimate strength and yield strength were maximum and decreased slightly at 1000°C and then again increased at 1020°C. This may be attributed to the precipitation strengthening effect of γ' and γ'' phase and dissolution of δ phase causing the grain boundary weakening effect with increasing solution treatment temperature [52].

It has been observed that the solution temperature of 980°C is not sufficient to completely dissolve Laves and δ phase and other microsegregated phases in additively fabricated Inconel 718. The Laves phase is partially dissolved at such temperatures leading to formation of local Nb rich areas enhancing the formation of δ

phase. This can be ascribed to the reduction in the amount of γ' and γ'' phase in the matrix. Therefore, solution treatment is carried out at a higher temperature [53].

It has been reported that homogenization should be carried out before solution treatment as beyond 1080°C, δ phase is not precipitated and Nb is uniformly distributed. Zhang et al. [22] reported without homogenization, the solution treatment would lead to the formation of local Nb enriched areas along with the formation of δ phase. On the contrary, if the solution treatment is followed by homogenization step, needle shaped δ precipitates are formed at the grain boundaries. The presence of δ precipitates at the grain boundaries enhances the creep strength by pinning down the grain boundaries and impeding grain boundary sliding [25,52]. As a result of high temperatures involved in the heat treatment procedure, the preferential $\langle 100 \rangle$ texture along the build direction thereby decreasing the anisotropy of the properties [22,25,52,54].

5.10 Double ageing

Ageing is a process in which the component is subjected to elevated temperatures for a considerable amount of time in order to increase the strength by producing precipitates of the alloying material within the metal structure [55,56]. The solution treatment is followed after by double ageing treatment to enhance the precipitation of the main strengthening phases of γ' and γ'' . With the presence of the strengthening phases in the metal matrix, the mechanical properties are also enhanced. Huang et al. [57] reported that the strength and ductility of AM Inconel 718 have increased after solution treatment followed by double ageing treatment.

The heat treatments individually have their own advantages but they always need to be carried out in the designed sequence in order to achieve desired properties. Pröbstle et al. [58] and Chlebus et al. [59] carried out direct double ageing treatment without carrying out the solution treatment. It was observed that γ' and γ'' were precipitated but the morphology of the interdendritic Laves was not changed. Therefore for more efficient precipitation of the γ' and γ'' during the ageing process, it must be followed after solution treatment (Table 5.2).

Huang et al. [57] observed that concentration of the Laves phase decreases with increase in the solution temperature but the grain size decreases with the increase. The tensile strength and ductility was increased after solution + double ageing heat treatment and exceeded the wrought Inconel 718.

5.11 Intrinsic heat treatment

During the LPBF process, the alloy experiences varying thermal profiles as it is subjected to subsequent processing steps. During the process, the metal powder is heated to temperatures above melting temperature followed by rapid cooling from this temperature. This is because of the small size of the melt pool relative to the

Table 5.2 The standard heat treatment procedure for Inconel 718 [60].

Standard	Treatment	Conditions	Cooling method
AMS 5663	Solution treatment; double ageing	980°C – 1 h 720°C – 8 h + 620°C – 8 h	Air cooling (AC) Furnace cooling (FC) at 55°C/h to 620°C air cooling
AMS 5383	Homogenization; solution treatment; double ageing	1080°C – 1.5 h 980°C – 1 h 720°C – 8 h + 620°C – 8 h	AC AC FC at 55°C/h to 620°C + AC
AMS 5664E	HIP; homogenization; double ageing	1180°C at 150 MPa – 3 h 1065°C – 1 h 760°C – 10 h + 650°C – 8 h	FC AC FC at 55°C/h to 650°C AC

size of the substrate or underlying layers, giving self-quenching effect. When the additional layers are deposited over or around the already deposited material in adjacent track or an overlaying layer, the adjacent material is in the heat affected zone of the melt pool. This is referred to as intrinsic heat treatment [61,62].

The temperature attained during the procedure is near melting temperature (remelting in some cases during the deposition of the ensuing layers) and drops quickly from layer to layer. The ability of intrinsic heat treatment to provide with secondary precipitation can allow in shortening or complete avoidance of subsequent ageing treatment.

Sasan et al. [63] reported that intrinsic heat treatment can be used to enhance the age-hardening response. The hardness and the yield strength was increased by triggering in situ nanoprecipitation. Seidal et al. [64] reported that intrinsic heat treatment is related to short high-temperature annealing. Barriobero-Vila et al. [65] reported that intrinsic heat treatment of Ti6Al4V introduced martensite decomposition due to higher exposure periods at higher temperature. Kurnsteiner et al. [66] investigated the effect of intrinsic heat treatment on Fe-19Ni-xAl and reported that the heat treatment activated the precipitation hardening mechanism in the alloy.

5.12 Suitable processing strategies

Due to the inherent nature of the AM process itself, LPBF process poses several challenges such as residual stresses, distortion, porosity, etc. in the fabricated component. Therefore utmost care should be taken during the selection of process parameters prior to printing to avoid losses.

It is observed that the laser energy density strongly influences the density of the part fabricated. The porosity in the parts increases with a decrease in the laser energy density until reaching the minimum required energy density required for melting. The VED has a statistical significance and can be used for process design and optimization. Watring et al. [28] suggested the use of laser energy density control to minimize the porosity and pore structure to optimize the mechanical properties of the fabricated component.

The island scan strategy has been reported to reduce the level of residual stresses developed in the component thereby reducing distortions. The island scan strategy allows faster heat dissipation due to smaller scan length, thus reducing the heat accumulation. This helps in reducing the thermal stresses generated and also reduces the processing time due to shorter scanning vectors.

The heat treatment procedure is aimed at maintaining the homogeneity in the microstructure along with the balance in the strengthening phases and relieving the inherent residual stresses as a result of the process. Homogenization followed by solution treatment above 1080°C allows the dissolution of the Laves and δ phase providing with the uniform distribution of Nb in the matrix. The creep strength is also enhanced due to the precipitation of needle shaped δ precipitates at the grain boundaries. Double ageing treatment (720°C – 8 hours + 620°C – 8 hours) then enhances the precipitation of the main strengthening phases, that is, γ' and γ'' phase, thereby enhancing the mechanical properties of the component.

5.13 Conclusion

LPBF is a complex manufacturing process dealing with extreme thermal gradients but provides exceptional control over the manufacturing capability over a variety of complex geometric shapes. In this work, the effect of laser processing parameters and heat treatment on the mechanical and microstructural characteristics of Inconel 718 alloy has been studied. The energy density's influence on part density and better microstructure must be further studied and a model can be used to interpret the effect of energy density input on the part quality. The effect of intrinsic heat treatment on the part quality and further heat treatment strategy must be studied using model aiming at viewing the effect of various scan strategies on the same.

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Design and topology optimization for additive manufacturing of multilayer (SS316L and AlSi10Mg) piston

6

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6.1 Introduction

Automobile engines are an engineering marvel that is characterized by its high power to weight ratios. An internal combustion-based automobile engine is the most common type of automobile engines widespread available today. The heart of an internal combustion engine is its piston, which is one of the major parts for converting the thermal-based energy to a mechanical one and is primary part that takes the high impact thermal load and converts it into a mechanical one. Owing to this reason, the piston always tends to have an alternating mass in nature and is constantly subjected to varying thermal load. The main purpose of the piston would be to transmit the mechanical forces generated by the thermal load to the connecting rod. It also serves other purposes such as heat dissipation of the combustion to the engine cylinder and to the cooling oil; proper sealing between the surface of the piston and the combustion chamber; and ensuring that there is no gas leakage or mixing of oil and fuel during regular functioning of the automobile.

For accomplishing the foresaid, the piston must be made of a material that could constantly withstand high temperatures without any impediments. Present live scale analysis reveals that the material that is being used for pistons is those of aluminum alloys. Aluminum holds out its advantages in terms that it has a lesser density and a higher rate of heat transfer that aids in having a lighter engine and in transferring the engine heat at a greater rate. However, as the rise of technology befalls, high performance engines come into scenario. These engines deal with a higher temperature and stress, which would not be clearly dealt with the standard aluminum pistons. Therefore, a change of material would seem to be inevitable but based on a thorough analysis, it could be understood no other material that is as economical, light weighted and whilst feasible to serve the purpose of efficient heat transfer is available.

Therefore, the prime motive was to look into other viable solutions to bring an effective change in the setup. Literature [1] and [2] suggest the usage of coatings

over the surface of the piston to overcome these drawbacks, but as technology develops, there are greater scopes of modification that could be applied to such parts to meet the requirements. One such least commonly used technique is that of the Additive Manufacturing. Techniques such as direct energy deposition and selective laser melting can efficiently help in making such layer with good geometrical accuracy and aid in efficient multimaterial compatibility. By using the traditional aluminum alloys for manufacturing such pistons and when they are coated with materials like those of stainless steel, it can aid in reducing the impact of thermal load at the interface where the temperature rise would be drastically high along the piston side, thus addressing the drawbacks of existing system without compromising the advantages [3].

6.2 Product design and development for additive manufacturing

Product design is an integral part of product development cycle, which is a wholesome term referring to the total life cycle assessment right from the initial phase of market analysis till the phase of the final product release. The product design phase has a huge impact on the entire product life cycle. A proper design can ensure proper functional life for the product; moreover, a colossal design would also take care of the product next generation of usage by including the design for reusability (which is not dealt in detail here). The planning phase of the product delineation and development is crucial for the growth and commercialization of the industry. Proper planning can make sure that the industry takes advantage of the current, and the future market opportunities and the manufacturer should focus on ways that can effectively develop strategies so as to transfer the product design to a reality in order to release the opportunities to the best possible extend. The goal of any manufacturer would be to make sure that the product is being produced with the most optimal design. The following figure depicts the steps involved in the additive manufacturing of any part. A well-planned model needs to be designed at the initial stage. The design needs to be done on the parameters that the product needs to satisfy ones it is released as a reality. A Standard Tessellation Language/STereoLithography (STL) file is generated on the model being designed, where every layer is sliced into thinner ones and is read as a new interface where every sliced continuum is read as a separate layer. The platform on which the build is to be constructed is prepared to depend on the type of process being used. Ensuing to which the part is being released as a reality as printing of each layer is done. Once the product is being constructed the build part is removed and from the platform of build and is postprocessed. The nature of postprocessing purely depends on the type of process and the type of the material being used. Prior to usage of the product, the parts are inspected for quality test and after the part is proved to be flawless, the product is used for live scale application [4] (Fig. 6.1).



Figure 6.1 Process of product design and development.

6.3 Design for additive manufacturing (DfAM)

Design for additive manufacturing is a field that blends the skills and arts of design with the field of manufacturing abetted by science in order to achieve manufacturability at an unprecedented scale as compared to that of traditional manufacturing. It is a complete modification of the traditional methodology of manufacturing where in a part of a larger dimension would be made, and in order to obtain the actual geometry, the unnecessary part has to be effaced. The traditional method of design collectively termed as DFMA, that is, design for manufacturing (DfM) and assembly, focuses on manufacturing and assembly processes that are to be done on the product. These techniques come along with its respective drawbacks like reduce design flexibility, part consolidation, increased period for releasing a product, and an increased complexity of the product. The deterrent in the traditional technique could be comparatively reduced in the newer technology of Additive Manufacturing, where parts that are being manufactured are released by cumulative addition of layers over one and other, thereby enabling one to create parts with nexus geometries. As there is a variation of the methodology there comes a dissimilitude of the design procedure, the new approach is a complete swerve of the traditional way of design. This gives rise to the newer area for design collectively known as Design for Additive Manufacturing (DfAM). The ideology behind this is to have maximized product performance through reconciliation of hierarchal structures, the size, and the shape, the material composition that can be brought under the capability of additive manufacturing; the following figure shows the steps in each of the process of DfM and DfAM. The

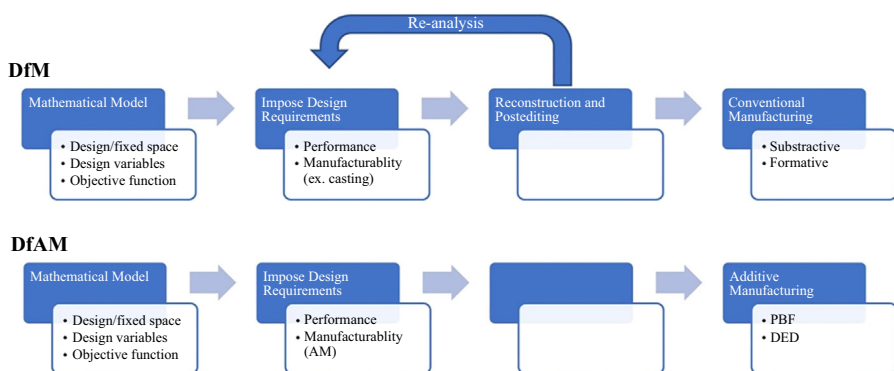


Figure 6.2 Comparison between the DfM and design for additive manufacturing. *DfM*, design for manufacturing.

ideology of DfAM negotiates the need of postediting and reconstruction thereby reducing the need of reanalysis and enabling the designer and the manufacturer to release the products at the earliest. It is important to note that part design and process design are directly linked together because they have a profound influence on each other, so they should not be considered in isolation. These ideologies lead to the opening of possibilities of having unique capabilities of having material, functional, shape, and hierarchical complexity [5] (Fig. 6.2).

6.4 Methodology and DfAM project design process for automotive piston

The part that is under study, that is, the piston, is habitually manufactured by using Computer Numerical Control (CNC)-based machining. The traditional material used for manufacturing is that of Aluminium alloys, which is built by casting and is later postprocessed to the final dimension. The postprocessing purely depends on the type of piston, which again indirectly depends on the type of engine with which the piston is being used; to cite an example for diesel engines, the top part of the piston requires the need of a piston bowl for ensuring homogenous mixing of air and fuel. A similar case could also be seen in the gasoline engine for GDI (gasoline direct injection), which requires a unique piston shape on the top to ensure that there is a swirl motion in the piston head so that there is ample mixing of inlet air and fuel. Therefore, the postprocessing, which needs to be done, would solely depend upon the type of piston and is presently carried out by traditional of CNC-based controller machines, which requires considerable time and money. However, as previously stated, the material of aluminum-based alloys needs to be modified due to rapid advancement in the technology, which demands the needs of material that is hefty in nature. Nevertheless, replacing such a part would prove out to be critical for engines as adding weight would indirectly reduce their performance. The work here pertains with dealing of such complexity, analyzing critically it could be understood that the top surface of the piston would be the part that is subjected directly to the thermal shocks and loads. Instead of making the entire component with a different material, it could be better if the top surface could be modified or coated with a newer material but proper measures should be made to make sure that the material being used at the top surface is compactable and is adherent to the surface at such high impact loads.

The current work deals with a manual-based design method rather than an analog-driven method owing to the fact that the work attempts to redesign the piston. The manual method is more preferred here because of the fact that most of the software that are being used for simulation-based design, such as those of topology optimization, need the same quantum of manual work after the simulation-based optimization in order to release the design for printing. When the redesigning of components for additive manufacturing is being done, the foremost step required would be redesigning the component using any Computer-Aided Drafting package.

This is followed the in next step where the principles of DfAM are applied. The parts to be manufactured are optimized in such a manner to have a reduced weight as when compared to traditional manufacturing AM provides more flexibility of manufacturing whose chances are to be explored into which can aid in reducing the weight of the part and the manufacturing time indirectly. The next and the most crucial step is to plan for manufacturing in such a manner that it has a lesser support structure and least printing time products with the least cost can be manufactured; moreover, the design must be altered without compromising the strength and function [6] (Fig. 6.3).



Figure 6.3 Concept of DfM and DfAM.

6.5 Generative design for additive manufacturing of automotive piston

CAD involves the development of designs. Given a set of constraints, many design alternatives can be created autonomously. Engineers and designers should use generative design to solve design problems in a more creative and effective way. By specifying goals and constraints, the generative design algorithm can study all possible design solutions, which provides engineers with multiple exploration options. The use of generative design in industrial applications has shown great promise, especially when combined with 3D printing. Simultaneous design generation and 3D printing will increase the versatility of the design while still producing lighter and stronger components.

Generative design (GD) and additive manufacturing (AM) are a perfect match, and they can complement each other perfectly. Each has its own advantages, but combined, they will be very powerful. According to the principle of generative design, varieties of design options for the same vehicle piston component are proposed. When creating a design, the location, load, and constraints must first be reserved according to project requirements. Automotive pistons have defined a topological definition suitable for evaluation. When redesigning existing components, automotive pistons will suggest a design scheme or several schemes. Based on the original design and manufacturing principles, generative design generates a large number of optimized designs for evaluation. In addition, in order to make wise choices, generative design uses previous experience to impose cost requirements for each result. Finally, the created design provides users with editable CAD-ready geometric models [7] (Fig. 6.4).

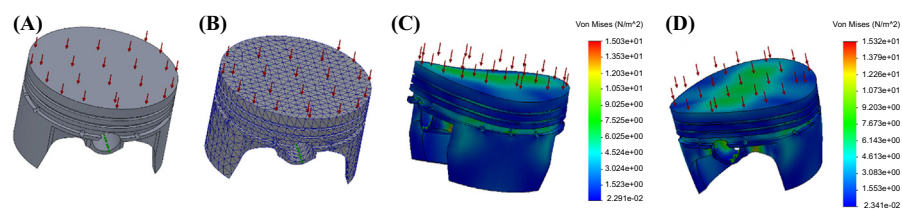


Figure 6.4 Automotive piston designed through generative design that can be additively manufactured.

6.6 Topology optimization for additive manufacturing of automotive piston

Topology optimization is an algorithmic method used to develop lightweight structures by removing materials from the design and predicting the most effective design based on a set of constraints or features. It is related to the number of related components/boundaries in the domain. Users can use topology optimization techniques to overcome the shortcomings of pure shape optimization.

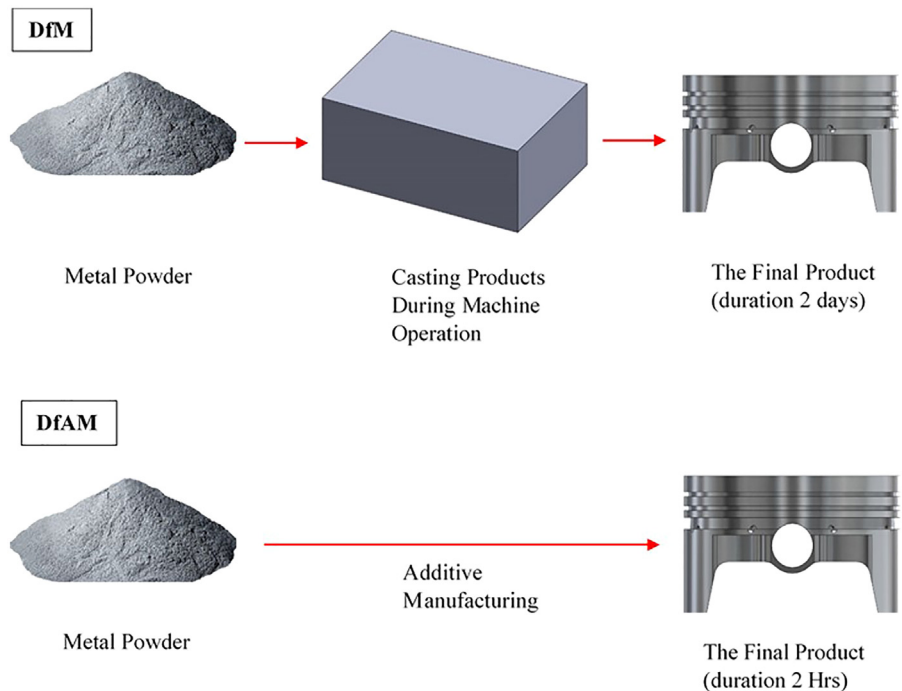


Figure 6.5 Topology optimization of automotive piston.

Topology optimization is usually carried out at the end of the design process, where material requirements need to be minimized without loss of part power. This is a statistical method that uses simulation techniques to predict the output without interpreting the simulation results. Topology optimization technology can automatically adjust component design to enhance functionality [8] (Fig. 6.5).

6.7 The automotive piston modeling techniques and simulation processes

Once the part design is completed with the aid of an analysis software, the strength of the part is determined. The analysis pertains to the extent to which the part would be able to carry load/stresses over its surfaces by considering ample material properties. These analyses can also help in reducing the material wastage in the component being manufactured [9] (Fig. 6.6).

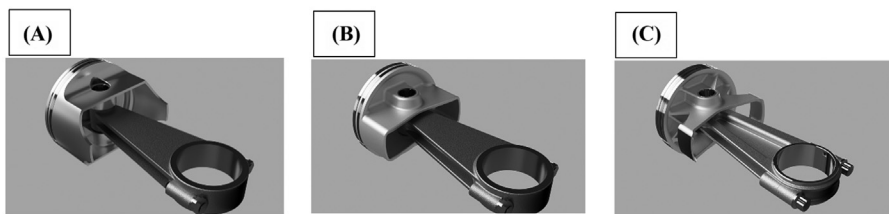


Figure 6.6 Automotive piston simulation processes.

6.8 Simulating additive manufacturing with additive software

An additive simulation software is indeed a unique and a powerful simulation tool for virtually releasing metal-based additive manufacturing. In the recent times, there have been a number of software tools available for predicting the outcomes of additive manufacturing right from printing to the end scale applications. These software enable the designer to study the residual stress that can develop during the printing of the part for any particular process, for example, Powder Bed Fusion (PBF); as there are variation in the input process parameters like scanning speed (mm/s), laser power (W), and layer thickness (μm), the corresponding values of stresses can be studied. Based on these values of simulation, the user could select the optimum values of input process parameter that can yield the least value or required value of stress. Such simulation can help manufacturers to reduce the cost and time involved in making of the components by reducing or eliminating the time incurred trial run process, thus enabling the users to reduce material wastage [10].

Additive-based simulation software features aid the end user to resolve many problems concerning 3D. Some of which are the following:

1. Obtain the most optimal build orientation
2. Minimize residual stress to avoid failure
3. Optimize the support structure that is used
4. Reduced distortion of the component that is built

6.9 Experimental optimization based on machine configuration

It can be evidently stated from research works that there is inherited connection between the process parameters used in additive manufacturing and their resultant mechanical properties, microstructure and their defect that are being generated during 3D printing of metal parts. From which it could be inferred that if improper printing conditions are used for processes, such as Powder Bed Fusion (PBF) and Direct Energy Deposition (DED), it could possibly yield to components with defects like voids, binding defects, and gas pores. There is a portent termed as balling that has chances of occurring in DED and PBF processes, and it depends upon the temperature of the molten powder particles and the solid surface. When the energy density of the laser is less, the balling phenomena may occur due to the improper wetting and incomplete melting characteristics of the powder, whereas higher laser energy density usage may increase the surface tension and change the molten characteristics. Improper scanning speeds may also result in the irregular size of the grains that are being build and can also affect the melt pool, which can change the quality of the parts by inducing pores. These porosities that form up in a part abetted by variation in the heating and cooling rate can also affect the micro-hardness of the component that is being manufactured. However, in the case of the DED process, the powder feed rate in addition to the laser energy density also would affect the characteristics of the component that is printed. For producing the part with the better quality, it is required to get the optimal combination of powder feed rate, and laser energy density as powder rate that is below par rate may cause crack generation and has very high possibilities of creating parts with improper fusion between particles. Hence, the optimal range of the parameters that are required for additively manufacturing the Stainless steel (SS316L) samples with the aid of the Powder Bed Fusion process is determined. EOS M280 machine is used for additive manufacturing of the sample of SS316L. The work here deals with the usage of Altair Inspire Print3D simulation software for determining the optimal combination of Powder Bed Fusion process parameter that can yield the minimal value of the residual stress without compromising on the maximum strength. The simulation trials were run with different laser power, layer thickness, and scanning speed from which the optimal combination of laser power, layer thickness, and scanning speed for part production were determined [11] (Fig. 6.7).

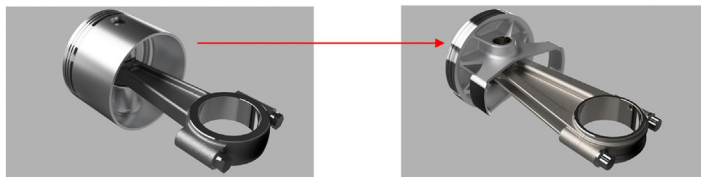


Figure 6.7 Process of powder bed fusion.

6.10 Part printing by a metal-based additive manufacturing process

6.10.1 Powder bed fusion

The powder bed fusion is a type of additive manufacturing technology where heat from a source is used to fuse powders that are spread on a bed as per the requirement of the part that needs to be manufactured. The different additive manufacturing techniques that fall under this category are selective heat sintering (SHS), selective laser melting (SLM), electron beam melting (EBM), selective laser sintering (SLS), and direct metal laser sintering (DMLS). Some of the most commonly used sources of heat are laser and electron beam, which are used to selectively heat the ingredients that are uniformly spread over the table at required places as per need. As a layer is deposited, the powder bed is indexed downward by the thickness of the constructed layer and a layer of powder would be deposited over the entire length. The powder spreading is generally done with the help of a roller or a blade, which deposits a uniform layer of the powder over the previous as-build layer.

The mechanical characteristics of the product that is being build is dominantly being influenced by the powder characteristics and printing parameters. A detailed study of the characteristics, such as microstructure, chemistry, and the morphology, is done by using X-ray photoelectron spectroscopy, laser light diffraction, differential thermal analysis, scanning electron microscopy, X-ray diffraction, and energy dispersive X-Ray spectroscopy. PBF can aid in processing a wide range of material alike high entropy alloys, superalloys (stellite, IN625, IN718), refractory materials (alumina, Ta-W, MoRe, Co-Cr), titanium alloys (γ -TiAl, Ti-6Al-4V), aluminum alloys (Al-Si-Mg, 6XXX, 7XXX), stainless steel (316, 316 L, 420, PH 17–4), tool steels (cermets H13), etc.

As stated previously, there are a couple of sources for selective melting of the powders to build the sample to the required dimension that is being constructed, out of which the laser-based PBF process is the most common used; the parameters influencing the characteristics of printed part are scan speed, laser power, remelting, scan pattern, over lapping ratio, hatch space, build chamber temperature, spot size, cooling rate, layer thickness, gas flow rate, laser pulse length, deposition mechanism, powder bed packing density, powder bed thermal conductivity, and energy density. These parameters may act as standalone parameters or can be a function of the others; for example, energy density of the laser can be said to be a function of

laser power, hatch space, scan speed, and layer thickness. It is worth notable that the parts that are being manufactured by the PBF process could possess some defects, which can be categorized as geometrical/dimensional related ones that include the size dimensional deviation, form dimensional deviation, etc.; surface quality defects that include balling, surface deformation, surface oxidation and surface roughness; microstructural-related defects that are due to the porosity, anisotropy, and heterogeneity; and mechanical properties-based defects such as fracture, poor bonding, cracks, holes, lower strength, and residual stress. The residual stresses that are induced during the PBF process have a considerable effect on the fatigue properties and the dimensional accuracy of the product being considered. Increasing scan speed, higher layer thickness, longer scan vector length, lowering beam power, base plate heating, and providing beam prescan/rescan are some proven techniques of reducing the net residual stress of the build component using PBF technique. A few other nonprocess oriented ways of reducing the residual stresses are annealing, hot isostatic pressing laser shock peening, and preheating.

One other type of PBF that is used is the electron beam based one, where an electron beam from a source is accelerated by a high voltage and selectively melts the powder thereby forming thin layers in a high vacuum environment. Since the electron beam is used, the energy density that is obtained is higher than the laser source, thereby causing a higher building rate as when compared to its laser counterpart. The powders that are used in the SLM process are preheated in order to improve the quality of the component being produced and also aid in reducing the unintended deformation. The accuracy of the EBM-based PBF is inferior to that of laser-based PBF because the layer thickness and the powder size used in the EBM are comparatively higher. EBM hold another critical drawback that in sense it is limited to the conductive materials. Due to its high operational cost, the EBM-PBF application is limited to the sectors of medical and aerospace. Some of the process parameters of EBM-PBF are beam voltage, the scanning rate, scanning sequence, layer thickness, number of contours, beam current, building orientation, focus, etc. EBM holds the upper hand in the PBF process because the residual stresses that are obtained in the EBM process are comparatively lesser than the SLM parts because as every layer is built, there is a stress relief annealing in EBM. The process of PBF offers advantages like capability of building functionally graded part, a better

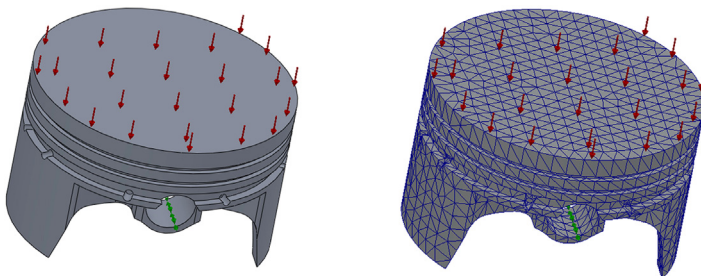


Figure 6.8 Process of direct energy deposition.

resolution, reduced material wastage, efficient recycling of unmelted powders, eliminating need of fixtures, better resolution of build, and accuracy. However, as the component is built in separate layers, the mechanical properties and the microstructure of the additively manufactured parts are unprecedented and in order to apply this phenomenon to live scale components, it requires detailed study of the same [12] (Fig. 6.8).

6.10.2 Direct energy deposition

Direct energy deposition is a classification of the additive manufacturing technique where the final geometry of the part is obtained by melting the initial raw materials that are fed in the form of wire or powder and control the deposition to obtain the required geometry. The melting of the geometry can be done by using any source of heat alike laser, electron beam or with the aid of plasma arc. There is a considerable difference in the efficiency of deposition as pertaining to the nature of material input form, if material of wire form when used as a feedstock displayed a greater deal of efficiency as compared to the material of powder form. DED over the duration of time has shown a wide variety of applications that include fabrication of entire parts, modifying the surface to meet new requirements and for appending material to the product that is already existing and/or repairing. The environment of fabrication using any particular technique of DED (the classification based on the source of heat) would depend on the type of the nature of the heat source as similar to the case of PBF that is based on electron beam; the DED that is based on electron beam would also demand a vacuum environment so as to avoid oxidation issues. Whereas in the case of powder DED, the powders are being blown off by the purging inert gas that is used to protect the melt region and also to aids in reducing the oxidation effect. The DED also provides flexibility to manufacture functionally graded material by using an attachment of an extra nozzle system. Since the powders that are being used in this case are of a larger size and since the energy density in the process is higher, the DED is combatively faster when compared to the PBF process, but the same happens with a reduced surface quality as compared to the PBF process. The process of DED also generally involves the usage of multiple axis turntables that minimize the need of support structures. The various techniques of additive manufacturing that are falling under this category are wire arc additive manufacturing (WAAM), laser engineered net shaping (LENS), and electron beam additive manufacturing (EBAM). Each of the foresaid DED techniques possess their own advantage and drawbacks. By using these techniques, it would be possible to repair products, achieve hybrid manufacturing capabilities, reduce material wastage, and develop multi material structure. Among these techniques, the usage of the wire arc and laser is predominate owing to its simplicity. The most optimal parameters are selected for developing parts with required characteristics because the mechanical properties of the final component that are built would be a function of these parameters. The variation of the heating and cooling rates would demand the need of postprocessing so as to produce part with better properties [13].

6.11 A case study of using additive manufacturing technology to manufacture automotive piston

The AlSi10Mg spherical powder used in the PBF process has a powder range of 20–63 μm and an average powder size of 32 μm . The chemical composition of AlSi10Mg powder is Al 88.8; Si 10; Mg 0.40; Cu 0.05; Fe 0.55; Mn 0.45; Zn 0.10; Ti 0.15; Ni 0.05; Sn 0.05; Pb 0.05. The DED machine uses SS316L spherical powder, the powder range is 50–150 μm , and the average powder size is 80 μm . The chemical composition of SS316L powder is Ni 10.59; Cr 16.99; Fe 68.66; Mn 1.4; Si 0.2; Mo 2.00; S 0.007; P 0.024; N 0.10; C 0.01. The EOS M280 machine uses the PBF method. The unit has a 400 W laser with a wavelength of 1060 to 1100 nm and is powered by a three-axis CNC. The process parameters of the PBF machine are laser power 350 W, layer thickness 30 μm , hatch line spacing 130 μm , and laser scanning speed 1650 mm/s. In the POM-DMD 105 machine is used, the laser power of the machine is 1 kW, powered by a five-axis CNC.

Fig. 6.9 shows the manufacturing unit where the DED process is performed on the PBF substrate to manufacture sandwich structure components. The process parameters of the DED machine are laser power, layer thickness, hatch line spacing, and laser scanning speed [14].

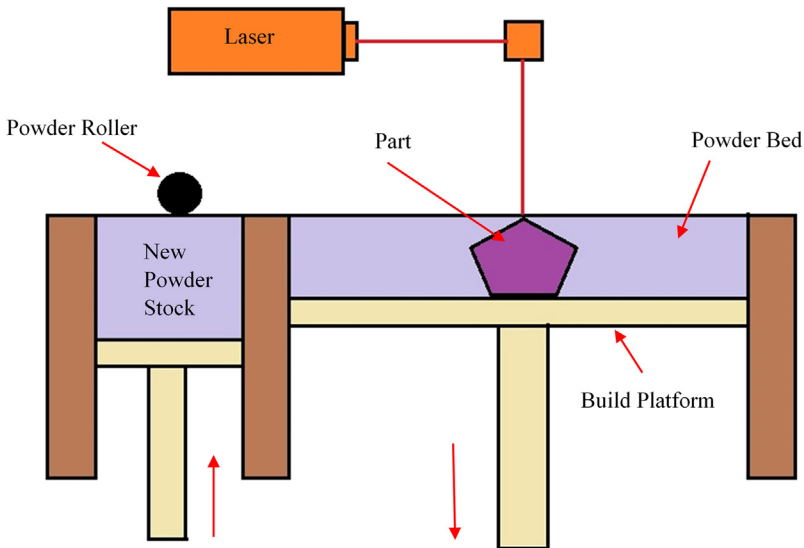


Figure 6.9 (A) DED 105D machine and (B) completed sandwich structure product.

Fig. 6.10 depicts the unique microstructure of the Sandwich structure, which was made on AlSi10Mg subtractive content (PBF) using DED. Both the optical microscope (OM) and the scanning electron microscope (SEM) are accurate and consistent, with no cracks or gaps. The heat-affected zone at the interface is very thin,

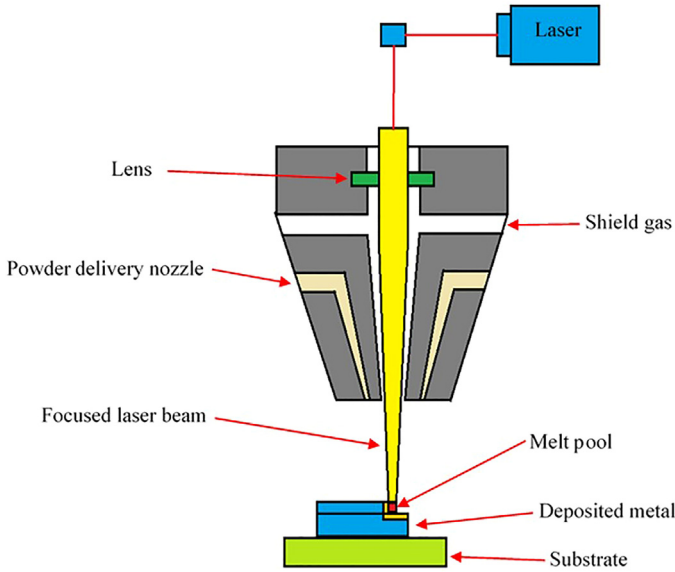


Figure 6.10 (A) Optical microscope image and (B) scanning electron microscopy image.

and the combination of the two processes is very good. The equiaxed grains formed above the interface indicate a faster cooling rate during DED. In addition, the epitaxial grain growth takes the shape of a heat flow path.

6.11.1 Numerical validation

According to Grashoff's formula, the thickness of the piston head (tH) is given by

$$tH = \sqrt{\frac{3PD^2}{16\sigma_t}} \text{ (unit in mm),}$$

where D is outside diameter or cylinder bore of the piston in mm = 50 mm; P is explosion pressure or maximum gas pressure in $\text{N/mm}^2 = 5 \text{ N/mm}^2$; and σ_t is permissible (tensile) bending stress for the material of the piston in N/mm^2 .

It may be taken as 275 N/mm^2 for SS316L

$$tH = \sqrt{\frac{3 \times 5 \times 50^2}{16 \times 275}} = 2.9 \text{ mm.}$$

Therefore, the thickness of the piston head, tH , is 2.9 mm (Fig. 6.11).

Fig. 6.12 shows the result of the Solidworks analysis value. The yield stress of the aluminum piston is 270 N/mm^2 , and yield stress of the sandwich structure is

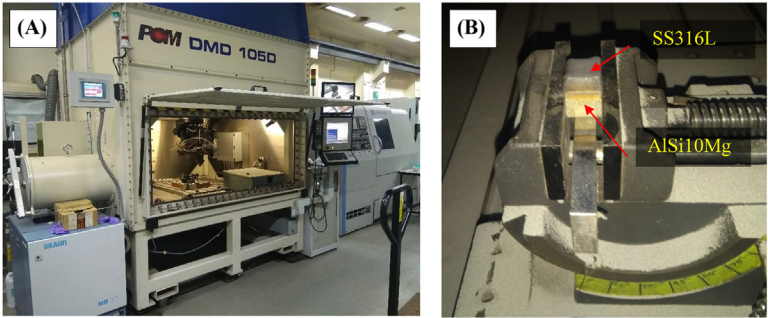


Figure 6.11 Construction of sandwich structure piston.

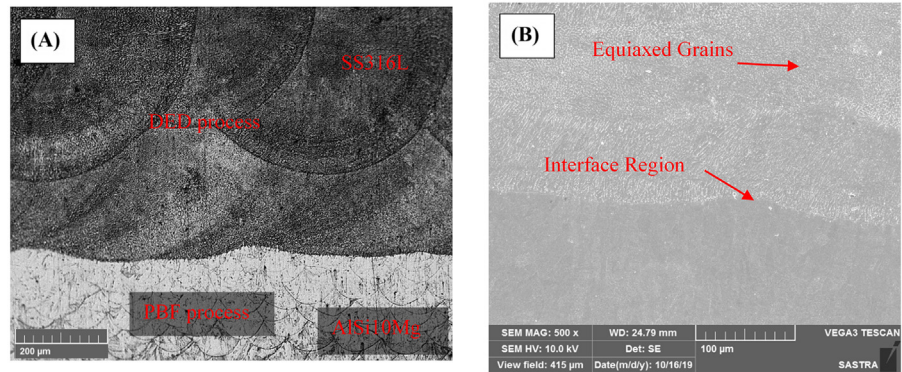


Figure 6.12 Solidworks analysis result: (A) loading condition, (B) meshing condition, (C) the regular piston analysis result, and (D) additively manufactured (sandwich structure) piston analysis result.

472 N/mm². Additively manufactured sandwich structure pistons can withstand greater loads.

6.12 Conclusions

In this study, a multimaterial configuration is introduced for the piston and the suggested sandwich structure is developed by the additive manufacturing process. By adopting AM techniques, the topologically optimized multimaterial piston can be fabricated in a short span. A sound metallurgical bonding is noticed between the additively manufactured SS316L and AlSi10Mg portion. Furthermore, the yield stress of the proposed sandwich structure is found as 472 N/mm², and it is higher than the yield stress value of the regular cast aluminum piston.

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Mechanical properties of titanium alloys additive manufacturing for biomedical applications

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Biomedical applications are designed and fabricated primarily by the requirements of the implant and are commonly used in different parts of the body. Therefore, the choice of materials is significant and needs to be prioritized. Such material should have the following advantages in the human body fluid environment, including great corrosion resistance, high strength, low Young's modulus, good wear resistance and no cytotoxicity. So far, three common metals have been used for implants, that is stainless steel, Co-based alloys and titanium alloys [1]. Titanium alloys have been extensively studied based on excellent mechanical properties such as lightweight, high strength, corrosion resistance, good biocompatibility and low modulus [2]. Conventional titanium alloy manufacturing methods such as casting and powder metallurgy need subsequent mechanical processing, consuming more time and energy.

Selective laser melting (SLM) is one of the powder bed fusion technologies based on the principle of melting stationary metal powder in a so-called powder bed, using a laser as an energy source [3] as shown in Fig. 7.1. After the first layer of the metal powder has been melted, the metal powder is again spread with special blades across the powder bed in a predefined thickness, and the melting process starts over again, in a layer-by-layer manner.

Product is formed by repeating this process of spreading and melting the metal powder. The titanium alloy's most researched and widely used conventionally and additively manufactured is Ti6Al4V. By alloying titanium with aluminium and vanadium, mechanical properties can be increased [6]. Systematic experimental data and knowledge on modelling monotonic and cyclic elastoplastic behaviour of SLM-ed titanium and its alloys are poorly available in the literature and very limited. Therefore, this chapter gives the mechanical properties of the most researched and widely used SLM-ed Ti6Al4V alloy, researched by different groups of authors. Further, a simple Ramberg–Osgood (R–O) material model is used on available experimental data to obtain monotonic and cyclic elastoplastic material parameters, which will serve as the first step for further comparison between this simple R–O

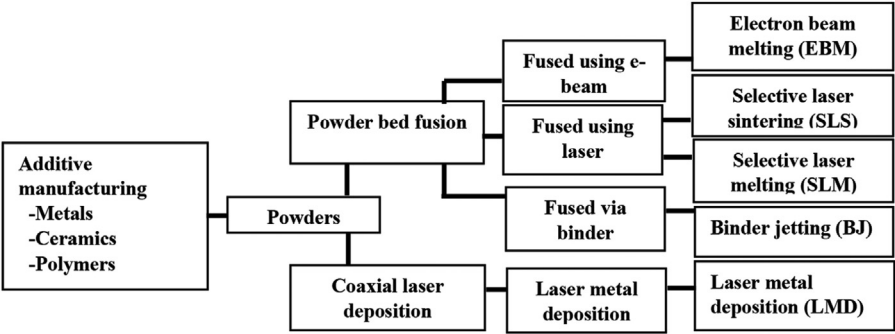


Figure 7.1 Additive manufacturing techniques for powders [4,5].

model and more advanced material models capable of capturing additional phenomena that may occur during cyclic loading [7,8]. Additive manufacturing (AM) concerns rapid prototyping manufacturing techniques based on successively stacking up several 2D layers of a material to form 3D objects with great precision.

The first patent on AM was filed in 1986 for a stereolithography technique [9]. In AM techniques, a virtual model prepared using a computer-aided design (CAD) software is fed into the AM machine that will ‘print’ the objects in well-defined dimensions, often precluding additional postprocessing steps [10,11].

Conventional manufacturing methods, on the other hand, require the removal of material (cutting/finishing/drilling) from bulk/sheet/standard commercial geometries and shapes to get the desired shape, which involves a high input of starting material and considerable material waste during postprocessing. AM enables the material to be printed strictly based on the specific design provided by the software and makes it easier to fabricate complex shapes, which may be difficult or even impossible to be manufactured by traditional methods [12,13]. For example, nozzles for fuel injection in spacecraft often involve complex structures made precisely by AM, which were previously manufactured by assembling several components [4,5,14]. The elimination of additional steps involved in assembling several smaller body components helps save substantial energy, labour, and production time. Hence, many lightweight structures that are applicable in several fields are today fabricated by AM on a commercial scale to reduce manufacturing costs and save time. In the field of medicine, dental and bone implants, as well as a variety of fixtures such as bone plates, screws, nails, and wires, are now manufactured by additive means, as the technique is capable of creating patient-specific components based on the patient’s medical imaging [15]. Similarly, for other industrial applications, AM of complex-shaped mixing and swirling burners has helped save much energy, increased components’ lifetime, and reduced the repair charge [16]. Various industrial hardware constituents such as punches, dies and other tools are rapidly manufactured by prototyping.

Among the newly developed AM processes that emerged during the late 1980s and early 1990s was the SLM technique applied to most nonvolatile metals. However, its

benefit is best realized when applied to metals that are not easily manufactured through utilizing other techniques [17]. Titanium alloys are one such group, possessing an array of exceptional properties, comprising high fracture toughness, outstanding strength-to-weight ratios, along corrosion resistance [18]. The high cost and poor machinability of Ti-6Al-4V utilizing conventional processing methods limit its more extensive application. Moreover, titanium alloy production using traditional processing tools results in high-energy consumption and extensive material waste.

Consequently, researchers are studying methods for processing titanium alloys using nontraditional technologies, such as AM. SLM powder AM process, which delivers greater benefits than traditionally applied production techniques, by decreasing the number of production steps, offering near-net-shape production combines improved level of flexibility and elevated material use efficiency [19]. Nonetheless, the unique conditions required for the SLM process cause certain problems, including but not limited to large thermal gradients emerging during the process, due to the limited interaction time and the highly localized heat input resulting in the build-up of residual thermal stresses. The rapid solidification causes the appearance of nonequilibrium phases and the segregation phenomenon. Additionally, the nonoptimal scan parameters may give melt pool instabilities during the process, resulting in higher surface roughness and increased porosity [20]. Bone tissue is arranged in a hierarchical structure. The submicrostructure contains lamellae consisting of layers of fibres of mineralized collagen organized in a planar form. Collagen fibrils are made of collagen molecules and hydroxyapatite (HA) mineral crystals [1]. Bone is a dynamic type of biological tissue that can continuously be reconstructed by two processes, modelling and remodelling, to keep it functional. The bone tissue is responsible for numerous vital purposes in the body, such as contributing to mineral equilibrium, being the primary section of haematopoiesis and contributing structural support for body and soft tissues [21]. Osteoblasts have played an important role in skeletal growth regarding many types of local, systemic, and mechanical stimuli that help mineralization while they organize bone remodelling. This cell is obtained from pluripotent mesenchymal stem cells (MSCs); they develop along a particular lineage to become extremely functional synthetic cells [22]. *Bone grafting* is a surgical operation that substitutes missing bone in extremely complex fractures with vital health risks.

7.1 Selective laser melting

Conventional fabrication routes are mostly subtractive processes that remove unwanted layers or edges, fixtures, and so on from a bulk body to obtain the desired shape. SLM is a powder-bed fusion process that adopts layer-by-layer powder melting using a high-energy laser beam(s) in an inert atmosphere, the melting sequence being under the CAD [17]. Fig. 7.2 illustrates the important components of the SLM machine. The 3D design is first sliced into several 2D layers of a defined thickness, often in 20–50 μm [25,26]. The powder distribution and melting are then

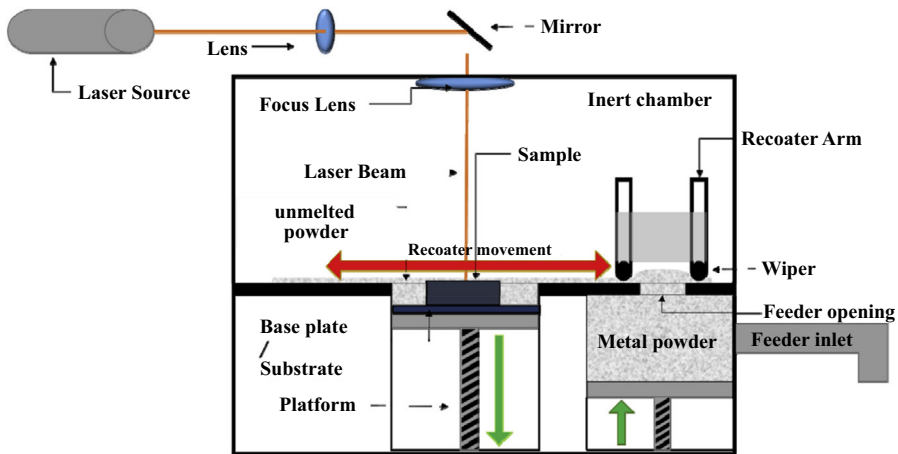


Figure 7.2 Basic components in a selective laser melting setup [23,24].

performed on a detachable platform inside the SLM chamber [21,22,25]. During the process, the powder is fed into the chamber with the help of a hopper feeder mechanism. A recoater blade or roller spreads the powder uniformly over the platform in the form of a thin layer. The laser is exposed to selected regions on the powder layer in conjunction with the design. After exposure, the platform moves down by a distance equal to the predefined layer thickness and the process of powder coating and laser exposure continues until the component is printed completely. The term ‘SLM’ thus indicates the melting of a selective/partial region on the powder bed. An SLM device’s main components are the laser source, the powder feeder, a computer system for instructions and control, an inert atmosphere in the processing chamber and a roller [27]. SLM-processed components may achieve full densification with minimal defects subject to the condition that the process parameters are carefully optimized [23,24,28,29]. Thus, the parameters need to be properly adjusted for every new material system [30]. One important aspect to be taken care of is that no mechanical pressure is applied on the powder layer during melting, but only gravity, capillary forces, and thermal effects contribute mainly to achieving densification upon solidification [31]. The relevant parameters that must be adjusted can be classified into two groups: powder characteristics and actual process parameters. The powder characteristics encompass material density, melting point, particle size, particle shape, powder flowability, laser reflectivity, thermal conductivity, melt pool viscosity and so on. Process parameters such as the laser wavelength and laser working mode are usually not varied but are determined by the device itself. The key to the successful manufacturing of reliable, defect-free components lies in optimizing the rest of the process parameters such as layer thickness, scanning speed, laser power, and hatch distance/style [32]. The laser energy density ‘E’ (defined as the amount of energy supplied to the powder bed) during the process is given by a study by Rao et al. [33].

The laser power itself strongly affects material characteristics such as density, morphology, and mechanical behaviour [34]. The laser power is mostly varied alongside the scan speed to determine the dwell time of the laser beam on the melt surface. The ratio of laser power (P) and scan speed (v) defines the linear energy density (J/m), which is a common and important parameter. From this relation, an increase in either laser power or a decrease in scan speed increases energy density. To clarify this, Eisenbarth and Breme [35] studied the effect of scanning speed and laser power on densification, microstructure, and properties of Inconel 718 superalloy. High scan speeds resulted in reduced dwelling time and reduced melt pool temperature, which attributed to poor melt pool dynamics and high porosity [36]. This high scan speed also resulted in coarsened microstructure and high residual thermal stress.

On the other hand, reducing the scan speed to a considerable value and increasing the laser power resulted in more heat generation and prolonged residence of the laser beam to attain optimum viscosity in the melt pool and achieve higher density. A finer microstructure can be obtained by optimizing the laser power and scan speed [37]. The laser beam moves over the powder bed at a predefined scan speed (v) during processing. Higher scan speeds result in shorter fabrication time and may also give rise to incomplete melting and defects [38]. However, the laser power may be increased sufficiently to avoid partial melting in some cases. The layer thickness is another important parameter directly proportional to the average powder particle size. Lack of coherence between the particle size and the powder layer thickness results in either over melting in case of too thin layer thickness or poor bonding between successive layers in case of too thick layers. The scan spacing (s), also called 'hatch space', that is, the separation between parallel laser tracks, is an important factor determining strong bonding between adjacent laser tracks within a given layer [39,40]. Adjacent hatches are generally made to overlap to avoid porosity and ensure high densification along with the layers [41]. Moreover, the laser beam can be tuned to traverse in several styles along with a layer. This defines the 'hatch style' or 'scanning strategy', which is essentially the pattern and length of the laser scanning vectors.

The 'manufacturing pattern', which is decided by the scanning strategy and scanning geometry, may be different for different materials or different purposes. The selection of an appropriate manufacturing pattern strongly influences the quality of the fabricated part. The laser scan patterns may involve straight and parallel lines with circular or spiral coverage. The variation in pattern direction can be changed inside a single layer or between successive layers. Different rotation angles of scanning are also possible between two layers. The densification and quality of SLM parts depend on the parameters above [42,43]. It is clear that the laser energy density can be increased either by increasing the laser power or by decreasing any or all of the parameters, including layer thickness, scan speed, and scan spacing. This increased energy density raises the temperature of the powder, and thus densification is achieved. Sufficient laser energy density is required to melt the layer to attain full densification completely. A minimum critical energy density is required for maximum densification [44]. For instance, the reported optimized critical laser

energy density for attaining fully dense (commercially pure, CP) Ti and Ti-6Al-4V is $\sim 120 \text{ J/mm}^3$ [45,46], and for Ti-24Nb-4Zr-8Sn, it is around 40 J/mm^3 [47]. Although the energy density seems to be a sufficient condition to obtain fully dense parts, it has been shown that the laser power is a more important factor and that each parameter defined in Eq. (1) needs to be optimized individually. This study reported the manufacturing of several parts by systematically varying the parameters in Eq. (1) so that the overall energy density was maintained at a constant value. The parts showed different microstructures and, hence, varying mechanical properties [48]. Another important parameter that directly affects the total energy input to the material is the shift in focus. Changing the focus means that melting the powder bed occurs at different heights concerning a fixed laser position [49].

7.1.1 Selective laser melting of titanium alloys

SLM can successfully produce a variety of titanium alloys, such as CP-Ti, Ti-6Al-4V and Ti-24Nb-4Zr-8Sn (Ti2448), and some of them have been well applied in the medical field [50]. CP-Ti is one of the most commonly used titanium alloys in AM printing medical applications as a traditional implant material. SLM as-produced CP-Ti samples mostly demonstrate the importance of manufacturing parameters, microstructures and mechanical properties [13]. It was reported that the input energy density (E) of 120 J/mm^3 is suitable to melt the powders sufficiently and build almost fully dense CP-Ti parts with a relative density is high than 99.5% [45]. However, the input power and scan speed should be adjusted at this energy density for achieving high-density parts (Fig. 7.3).

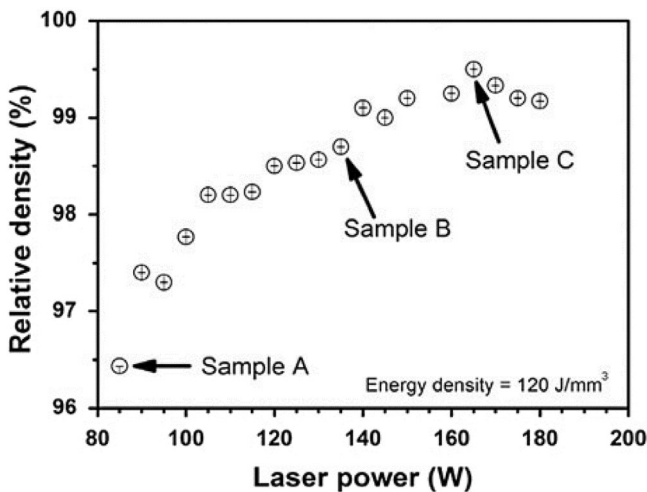


Figure 7.3 Relationship between relative density and laser power for the SLM-fabricated CP-Ti parts at fixed energy density of 120 J/mm^3 . Samples A, B, and C show different examples of relative densities [11].

7.2 Electron beam melting

Electron beam melting (EBM) is another AM system equipped with an electron beam launching device, which can produce nearly full density parts in a vacuum environment [51]. The working principle and EBM process are similar to that of SLM (Fig. 7.4) [47,51]. The main difference with SLM is that EBM uses a different heat source and chamber atmosphere. The heat source of EBM is an electron beam with a voltage of 60 kV, which preheats the substrate plate to a presetting temperature before dropping the powder [50]. The electron beam will prescan the powder for sintering and then scan the powder bed based on the sample CAD geometric shape. These differences in beam energy input and chamber environment of EBM and SLM result in the different microstructure (referred to melt pool size, phase-type, and grain size) and mechanical properties (referred to hardness, compressive, tensile, and fatigue properties) of SLM and EBM products. The densification rate and microstructural homogeneity of the EBM-produced part with an optimized parameter improve relative density and mechanical properties [45,46]. Some studies have been conducted to study the performance of EBM as-fabricated components and improve the properties of those samples.

7.2.1 Biocomposites materials reinforced with multiwalled carbon nanotubes

Bone tissue is arranged in a hierarchical structure. The submicrostructure contains lamellae consisting of layers of fibres of mineralized collagen organized in a planar

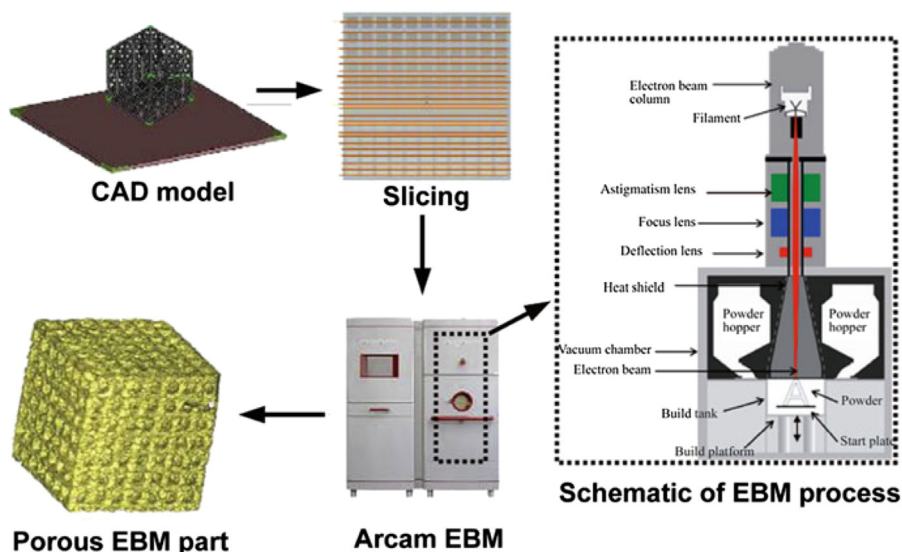


Figure 7.4 Schematic diagram of electron beam melting system [24,28].

form. Collagen fibrils are made of collagen molecules and HA mineral crystals [51]. Bone is a dynamic type of biological tissue that can continuously be reconstructed by two processes, modelling and remodelling, to keep it functional. The bone tissue is responsible for numerous vital purposes in the body, such as contributing to mineral equilibrium, being the primary section of haematopoiesis and contributing structural support for body and soft tissues [42]. Osteoblasts have played an important role in skeletal growth regarding many types of local, systemic, and mechanical stimuli that help mineralization while they organize bone remodelling. This cell is obtained from pluripotent MSCs; they develop along a particular lineage to become extremely functional synthetic cells [50]. *Bone grafting* is a surgical operation that substitutes missing bone in extremely complex fractures with vital health risks to the patient or fails in the healing process. This treatment method is used in numerous dysfunction cases, including trauma-associated osseous defects, delayed fusion or nonfusion of fractures, infection, congenital pseudoarthrosis, tumours, and facial reconstruction surgery [43]. The main disadvantage of bone grafting is that the harvest from the place is often extremely painful, especially after the operation and has a vital risk of increasing complications such as infection, haematoma, nerve injury, in some cases leaving an area of numbness near in harvest region and donor risks [44]. Biomaterial, an impressive field with strong development over its approximate half-century presence, contains parts of materials science, medicine, chemistry and biology [48]. After implantation, bone biomaterials serve as a medium for the interaction and contact of bone implants with the enclosing tissues/cells. So, bone biomaterial choice is an important step in forming ideal bone implants. Bone implants should possess mechanical characteristics that match the natural bone in the injury site and newly formed tissues through bone repair stages. Otherwise, failure may occur in bone repair inside the human body. The mechanical properties of selective bone biomaterials are deemed one of the most significant selection criteria [45]. Many biomaterials such as ceramics, synthetic polymers, natural polymers, metals, and composites have been widely applied in biomedical applications. The selective material pattern performs a significant function in the properties of bone scaffolds.

7.3 Electron beam melting of titanium alloys

EBM technology could process titanium components under a vacuum environment and obtain lower costs and more efficient production than conventional methods. Moreover, the advantages of EBM include the ability to produce complex shapes to meet specific industry needs and other advantages such as short processing cycles and efficient material utilization, making it an excellent alternative to titanium products. So far, Ti-6Al-4V alloy has been widely used for EBM production. In order to improve the mechanical properties of the resulting components, extensive efforts have been made to optimize the process parameters to manipulate the microstructure of EBM-produced samples. Researches have also been conducted on another

type of titanium alloy such as β -type Ti2448 [5,30]. The microstructure of EBM as-produced Ti-6Al-4V is very complicated. The EBM process is a promising additive manufacturing technique for metal parts, in the electron beam shots at high speed to the powder bed and melts the powders to generate a melt pool, and the liquid metal rapidly solidified. As the building layers accumulate, the solidification of the solid part of the building below the building level is affected by the multiple thermal cycling effects in the vacuum environment due to lower cooling rates and heat build-up in the EBM process. This results in the overall temperature of the sample being maintained at a high level, which plays a key role in stress annealing elimination. Such a process facilitates the good matching of sample strength and plasticity and uniformity of part performance [5,9,45,50]. The solid part of EBM as-produced Ti-6Al-4V contains columnar prior β grains, which are delineated by α grain boundary and a transformed α/β structure, and Widmanstätten pattern and lamellar colony within the prior β grains [14,40]. The generation of an α grain boundary along the grain boundaries of the prior β grains indicates the diffusive nature of the β - α transformation. In this way, the microstructure obtained in the EBM-produced sample differs from that observed in other AM-produced parts. Such as in the SLM process. The EBM as-produced porous structure contained α phase due to a high-temperature building environment and long time for cooling down. Liu et al. reported that the EBM-produced Ti2448 rhombic dodecahedron porous structures with a porosity of 70% exhibited great mechanical properties; for example, the ductility and the strength of the EBM as-produced sample are $\sim 11\%$ and ~ 37 MPa, respectively. The Young's modulus is only ~ 0.86 GPa, much lower than the Ti-6Al-4V rhombic dodecahedron porous samples with the same porosity (~ 1.4 GPa) [47]. EBM-processed Ti2448 porous samples have about two times the strength-to-modulus ratio of the Ti-6Al-4V porous structures with the same structure and porosity of 70% [9]. Fig. 7.5 shows the side view of EBM-processed, X-ray diffraction patterns and microstructure.

7.4 Conclusion

This chapter summarizes the development of biomedical titanium for SLM and EBM. Biomedical titanium alloys such as Ti-6Al-4V are generally preferred for medical implants because of their low Young's modulus, superior biocompatibility, and high corrosion resistance compared to stainless steels and CoCr alloys. The porous structure can further improve the biocompatibility and reduce Young's modulus of titanium alloy. Ti-24Nb-4Zr-8Sn (Ti2448) and other new biomedical titanium alloys with low modulus and nontoxic components will be the promising material of choice for the next generation of biomedical implants. AM technology based on titanium biomaterials has great potential in the medical industry. The complex structure made by AM provides enhanced mechanical properties and improved bone in-growth in the manufacture of artificial implants. Further research needs to focus on improving the roughness of AM implants by surface treatment and reducing defects by optimizing the building

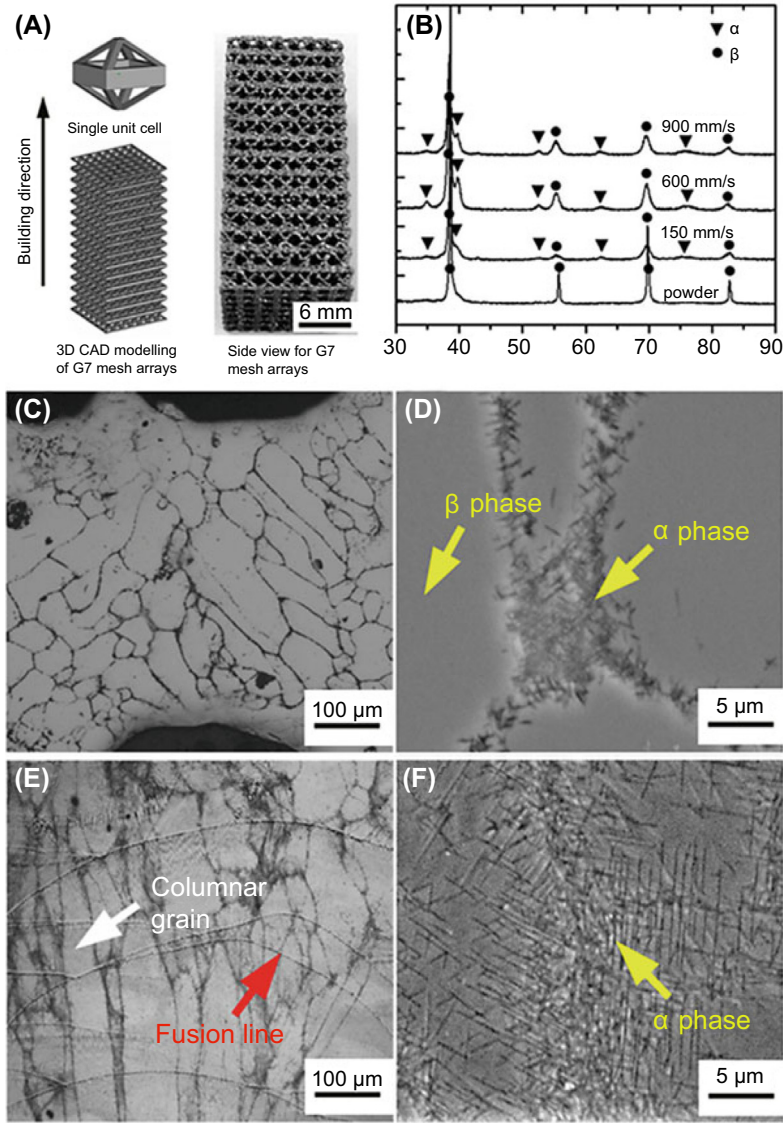


Figure 7.5 (A) Porous structure model used and side view of EBM-processed Ti2448 component, (B) XRD profiles of the starting powder and EBM-processed components, (C) and (E) OM, and (D) and (F) SEM images of Group A. Parts (C) and (D) are at horizontal plane and Parts (E) and (F) are vertical views [8].

process. More studies are needed to prepare and fabricate for porous gradient structures with gradient Young's modulus to reduce the implants' stress concentration and design and manufacture more reliable implants that are more suitable for the human body and bring better clinic results for patients.

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